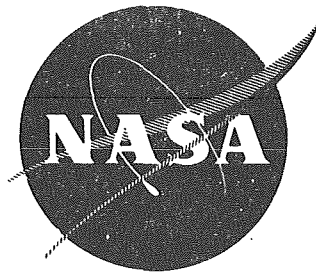


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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT NO. 12

prepared by
R. W. Harrison

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-6474
Robert L. Davies, Project Manager

NUCLEAR SYSTEMS PROGRAMS
SPACE SYSTEMS
GENERAL  ELECTRIC
CINCINNATI, OHIO 45215

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June 14, 1968

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NASA Lewis Research Center
Cleveland, Ohio
Robert L. Davies, Project Manager
Materials Section

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FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. R. L. Davies of NASA - Lewis Research Center is the NASA Technical Manager.

The program is being administered for the General Electric Company by E. E. Hoffman, and R. W. Harrison is acting as the Program Manager. J. Holowach, the Project Engineer, is responsible for the loop design, facilities procurement, and test operations. Personnel making major contributions to the program during the current reporting period include:

Alkali Metal Purification and Handling - Dr. R. B. Hand, L. E. Dotson, and H. Bradley.

Welding and Joining - W. R. Young and S. R. Thompson.

Partial Pressure Gas Analyzer Calibration - Dr. T. F. Lyon.

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from January 15, 1968, to April 15, 1968. The primary task of this program is to fabricate, operate for 10,000 hours, and evaluate a T-111 Rankine System Corrosion Test Loop. Materials for evaluation include the containment alloy, T-111 (Ta-8W-2HF), and the turbine candidate materials, Mo-TZC and Cb-132M, which are located in the turbine simulator of the two-phase potassium circuit of the system. The loop design will be similar to the Cb-1Zr Rankine System Corrosion Test Loop; a two-phase, forced-convection, potassium corrosion test loop which has been developed under Contract NAS 3-2547. Lithium will be heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium loop will be accomplished by radiation in a high-vacuum environment to the water-cooled chamber. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, namely:

- a. Boiling temperature, 2050°F;
- b. Superheat temperature, 2150°F;
- c. Condensing temperature, 1400°F;
- d. Subcooling temperature, 1000°F;
- e. Mass flow rate, 40 lb/hr;
- f. Boiler exit vapor velocity, 50 ft/sec;
- g. Average heat flux in plug (0-18 inches), 240,000 BTU/hr ft²;
- h. Average heat flux in boiler (0-250 inches), 23,000 BTU/hr ft².

In addition to the primary program task cited above, the program also includes capsule testing to evaluate advanced tantalum alloys of the ASTAR 811 type (Ta-8W-1Re-1Hf) in both potassium and lithium.

Also included in the program are the fabrication, 5000-hour operation, and evaluation of a 2600°F, high-flow velocity, pumped-lithium loop designed to evaluate the compatibility of the ASTAR 811-type alloys, T-111, T-222, and the tungsten alloy, W-25Re-30Mo.

II. SUMMARY

The instrumentation and installation of the T-111 Rankine System Corrosion Test Loop was completed.

The test loop was filled with alkali metal and the low-power, all-liquid pretest operation, which includes the checkout and calibration of the loop instrumentation, was completed.

Boiling and condensing operation was initiated, but the design operating conditions were not reached due to a leak in the potassium boiler. The loop was drained of alkali metal, and plans are now being prepared for the repair of the boiler.

The EM pump and surge tank subassemblies of the 2600°F Lithium Loop have been fabricated.

III. PROGRAM STATUS

A. T-111 RANKINE SYSTEM CORROSION TEST LOOP

1. Instrumentation and Installation of the T-111 Corrosion Test Loop

The T-111 Rankine System Corrosion Test Loop is shown in Figure 1 after the loop thermocouples were installed but before the loop was transferred from the clean room to the vacuum chamber system. The locations and designations of the 70 thermocouples which were installed on the loop are shown in Figure 2.

A typical thermocouple circuit originates at the hot junction of the thermocouple as shown in Figure 3 for the tube-in-tube boiler thermocouples. The thermocouple leads are routed along the support structure to a thermocouple reference junction block attached to the inside wall of the spool section. The thermocouple reference junction block shown in Figure 4 consists of a 99.7% Al_2O_3 terminal strip mounted on a copper block and mechanically fastened to the walls of the chamber. The entire assembly is shielded from the loop to minimize temperature gradients in the junction block as well as maintain a lower absolute temperature approaching that of the water-cooled vacuum tank wall. At the reference junction block, a transition from the thermocouple wire to copper wire is made, and the copper wires are routed through the thermocouple vacuum feedthroughs (shown in Figure 5) to three 24-point recording potentiometers. The temperature of the thermocouple reference junction block will be measured by W-3Re/W-25Re thermocouples and will be electrically connected to the recording circuit as shown in Figure 2 to automatically compensate for variations in the reference block temperature during operation.

Thermal insulation consisting of multiple layers of Cb-1Zr foil was simultaneously applied to the loop as the thermocouples were installed. The insulation used on all circular pipe sections was 0.002-inch thick x 0.5-inch wide foil which had been dimpled by passing the foil between a hardened-steel, coarse-knurled roller working against a hard plastic sheet. The effective thickness of the foil after dimpling was between 0.009 to 0.012 inch. The insulation was attached to the tube by spot

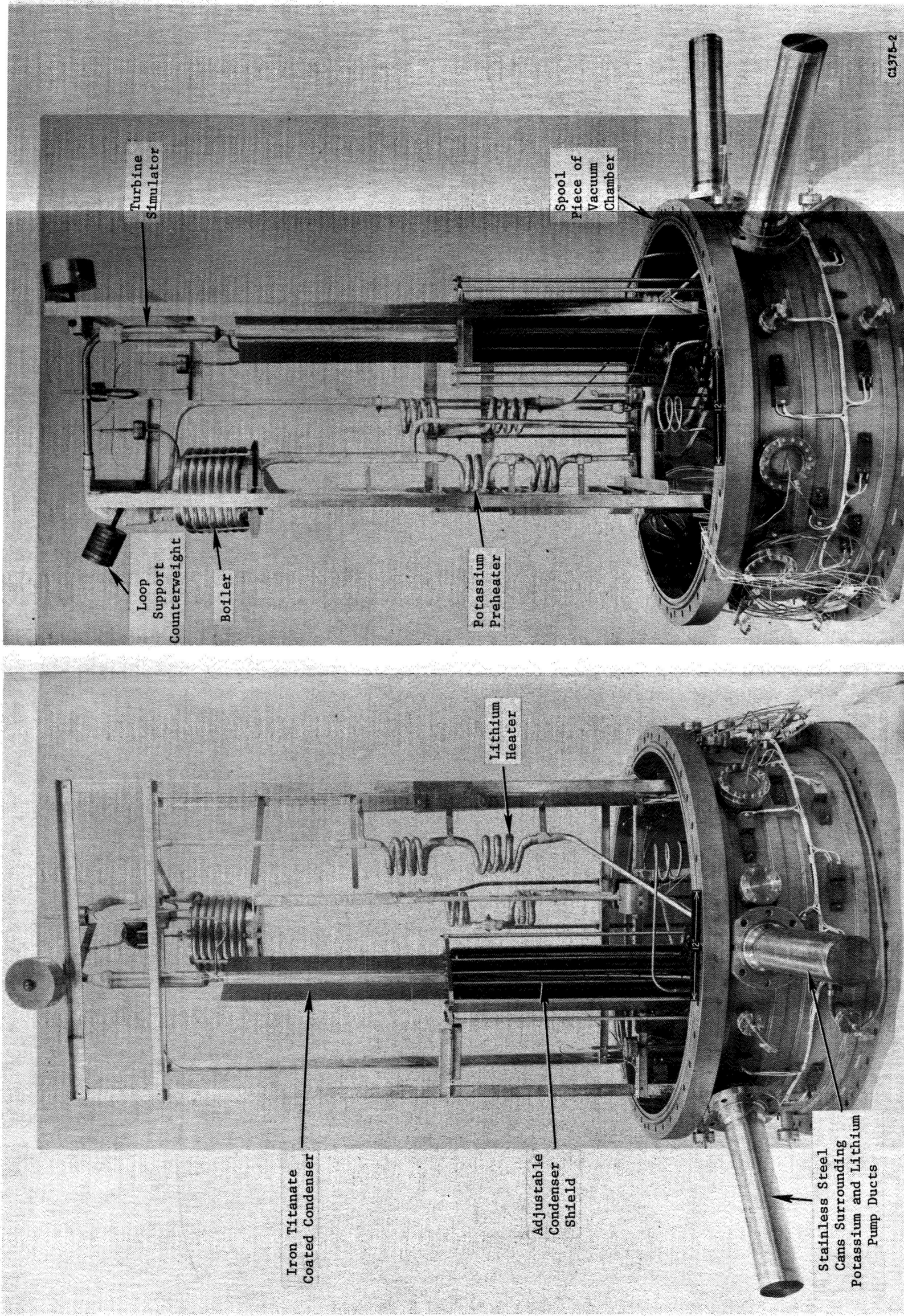


Figure 1. T-1111 Rankine System Corrosion Test Loop. Following Instrumentation Before Installation on the Vacuum Chamber Sump.
(Orig. C68013187, C68013190)

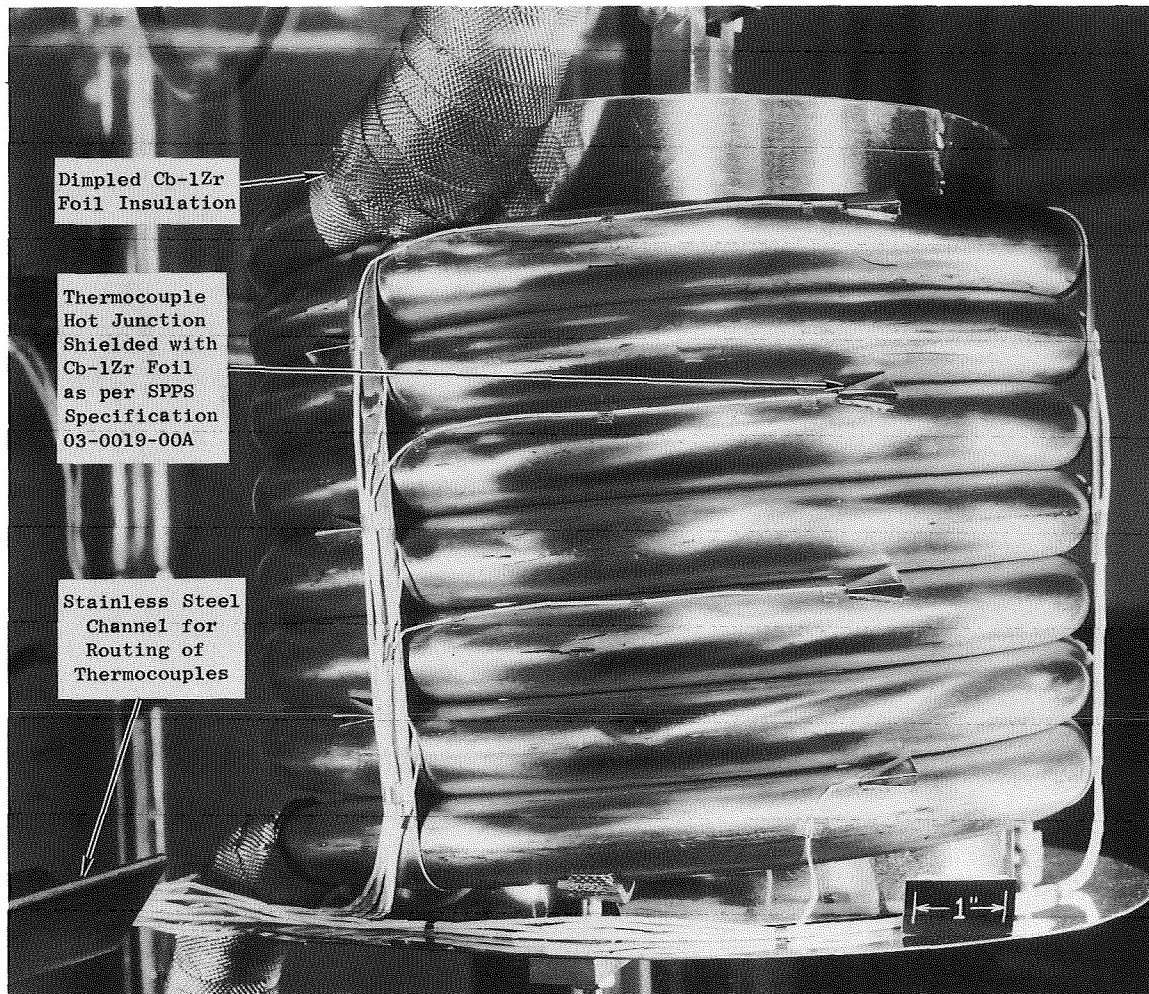


Figure 3. Thermocouple Installation on the Tube-in-Tube Helical Boiler of the T-111 Rankine System Corrosion Test Loop. (Orig. C68013186)

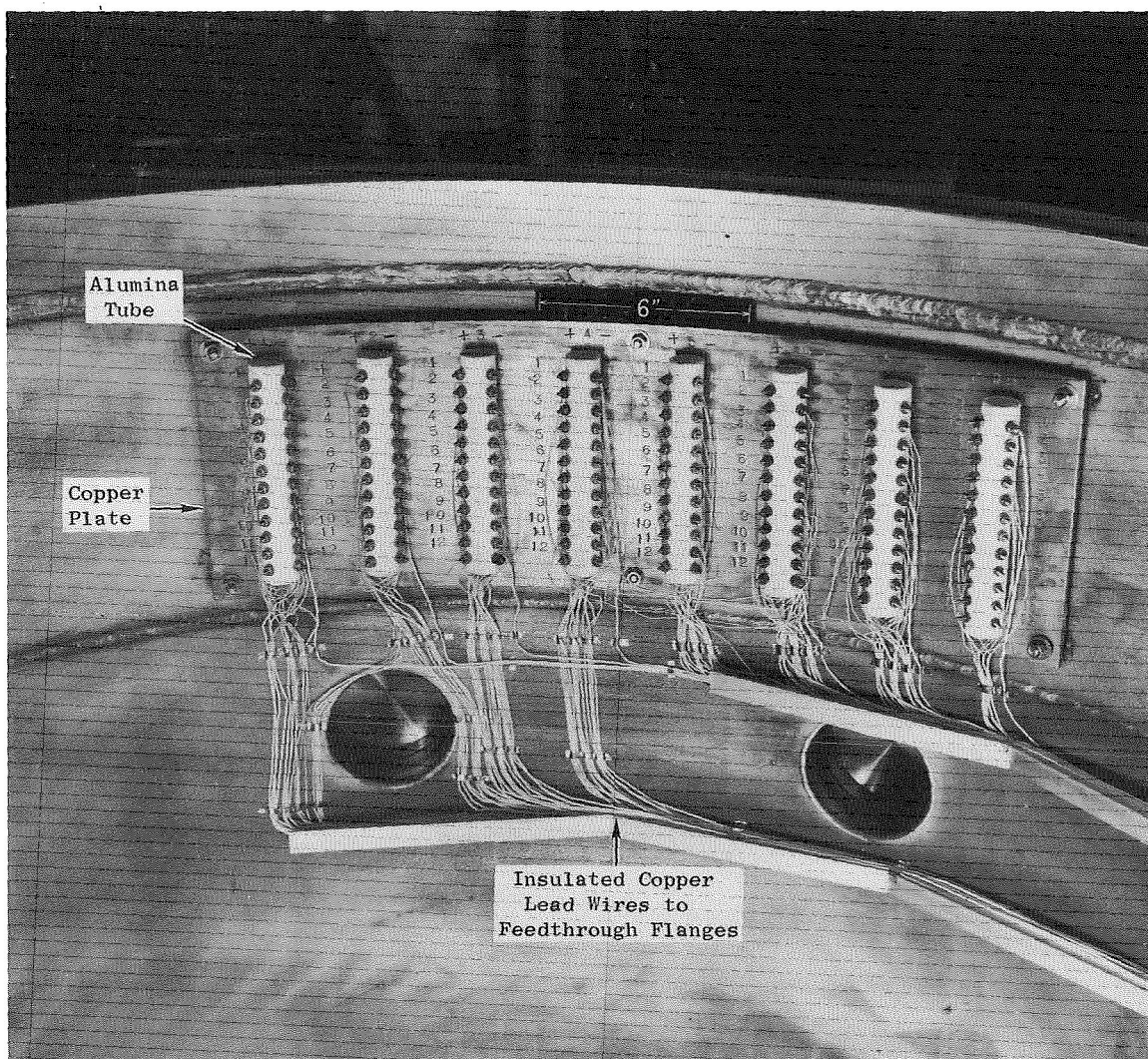


Figure 4. Thermocouple Wire Terminals (192 Total) Located in the Sump of the Test Chamber for the T-111 Rankine System Corrosion Test Loop. (Orig. C65062196)

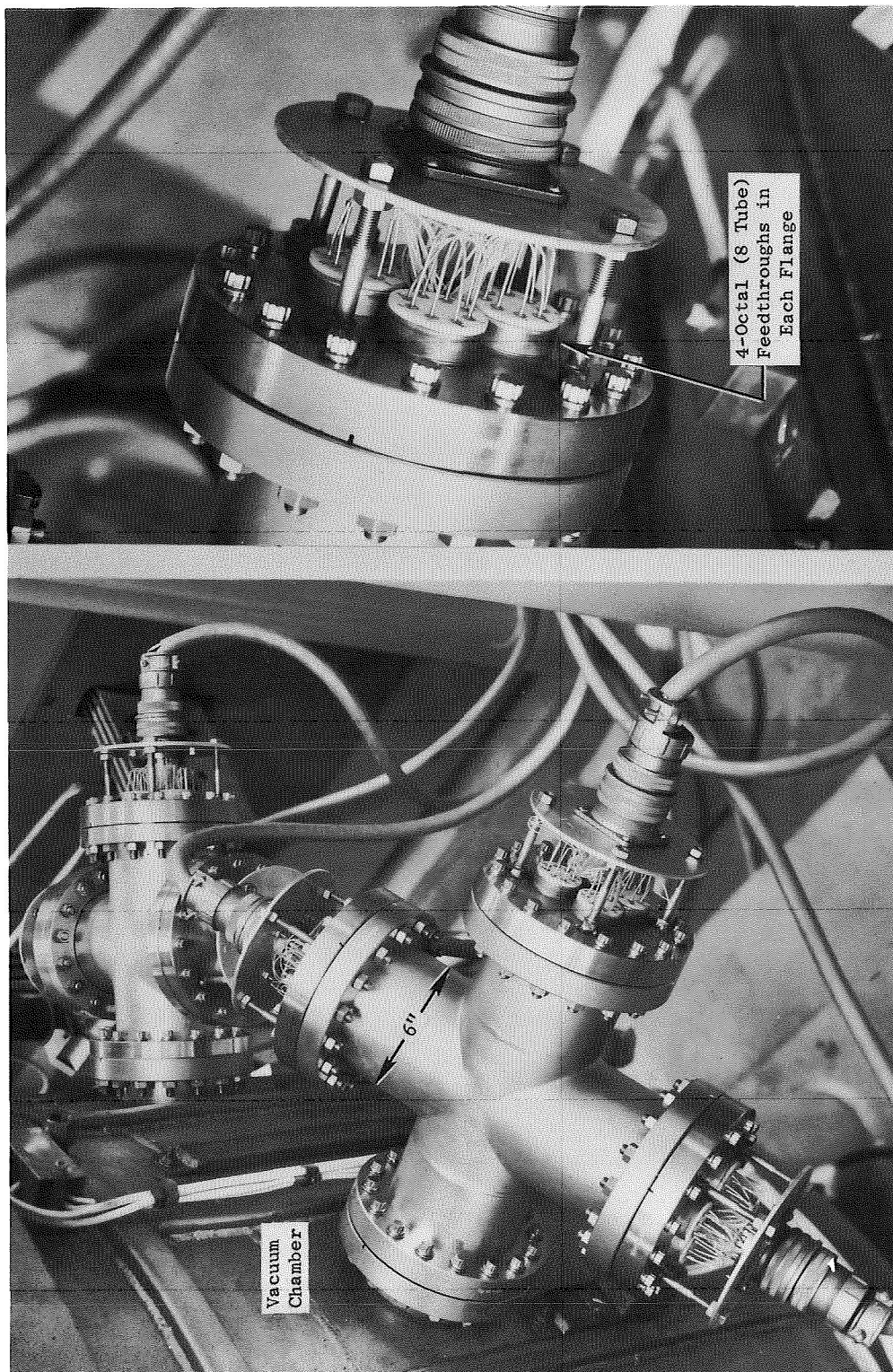


Figure 5. Six Thermocouple Lead Wire Feedthrough Flanges with 32 Wires in Each Flange. An Enlarged View of One of the Flanges is Shown on the Right. (Orig. C65062189)

welding the foil to the tube and to itself as succeeding layers were applied as shown in Figure 6. A minimum number of spot welds were used to minimize conduction heat losses through the foil. A molybdenum electrode was substituted for the copper spot welder electrode to avoid contamination of the foil surfaces with copper and an argon cover gas used to protect all welded areas from oxidation.

The loop is shown in the clean room after completion of the instrumentation and insulation in Figure 7. The plywood pallet on which the spool section and loop rested was lifted by a fork truck and held above the vacuum sump while the bell jar was lowered and then bolted to the spool flange. The bell jar and the attached spool section were raised together off the pallet and then lowered to the sump following removal of the pallet. The flanges were bolted together, and the vacuum chamber was evacuated and helium leak checked. This sequence was required to ensure a leak tight joint between the sump and lower spool flange because the spool becomes fixed in position when the EM pump windings for the two pumps are attached to their spool flanges and the gas pressurization and liquid metal fill lines are welded to their spool piece ports.

Upon completion of the helium leak check, the vacuum system air release valve was opened and the chamber was returned to atmospheric pressure. The bell jar was hoisted into the penthouse above the chamber, and the final instrumentation and the installation of loop components, which could only be completed with the loop in its final test position, were resumed.

The metering and on-off isolation valves, as shown in Figure 8, were assembled with their actuating systems which consist of ultra-high vacuum rotary feedthroughs^(a) with a torque rating of 6 ft-lbs connected by a 0.31-inch diameter flexible stainless steel cable to a 3:1 right angle gear drive mounted on the valve yoke.

A 500-watt quartz lamp heater,^(b) shown in Figure 9, was installed on the shell of the lithium surge tank to supplement the vacuum chamber

(a) Varian Associates, Model No. 954-5039.

(b) General Electric Model 500T3CL, 117 V

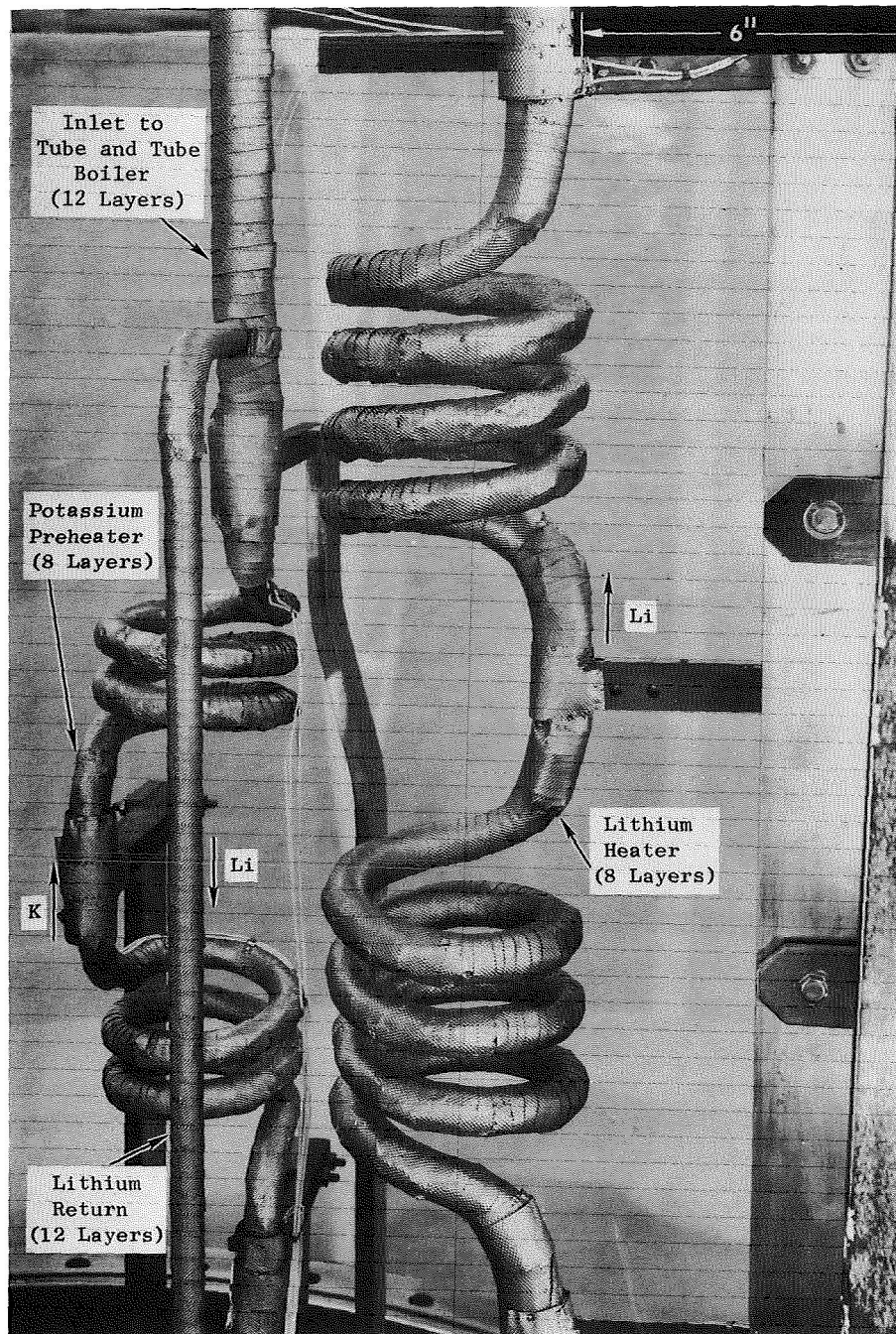


Figure 6. Typical Cb-1Zr Dimpled Foil Thermal Insulation of Various Loop Components. (Orig. C68013191)



Figure 7. T-111 Rankine System Corrosion Test Loop Following Instrumentation in the Clean Room. (Orig C68013197)

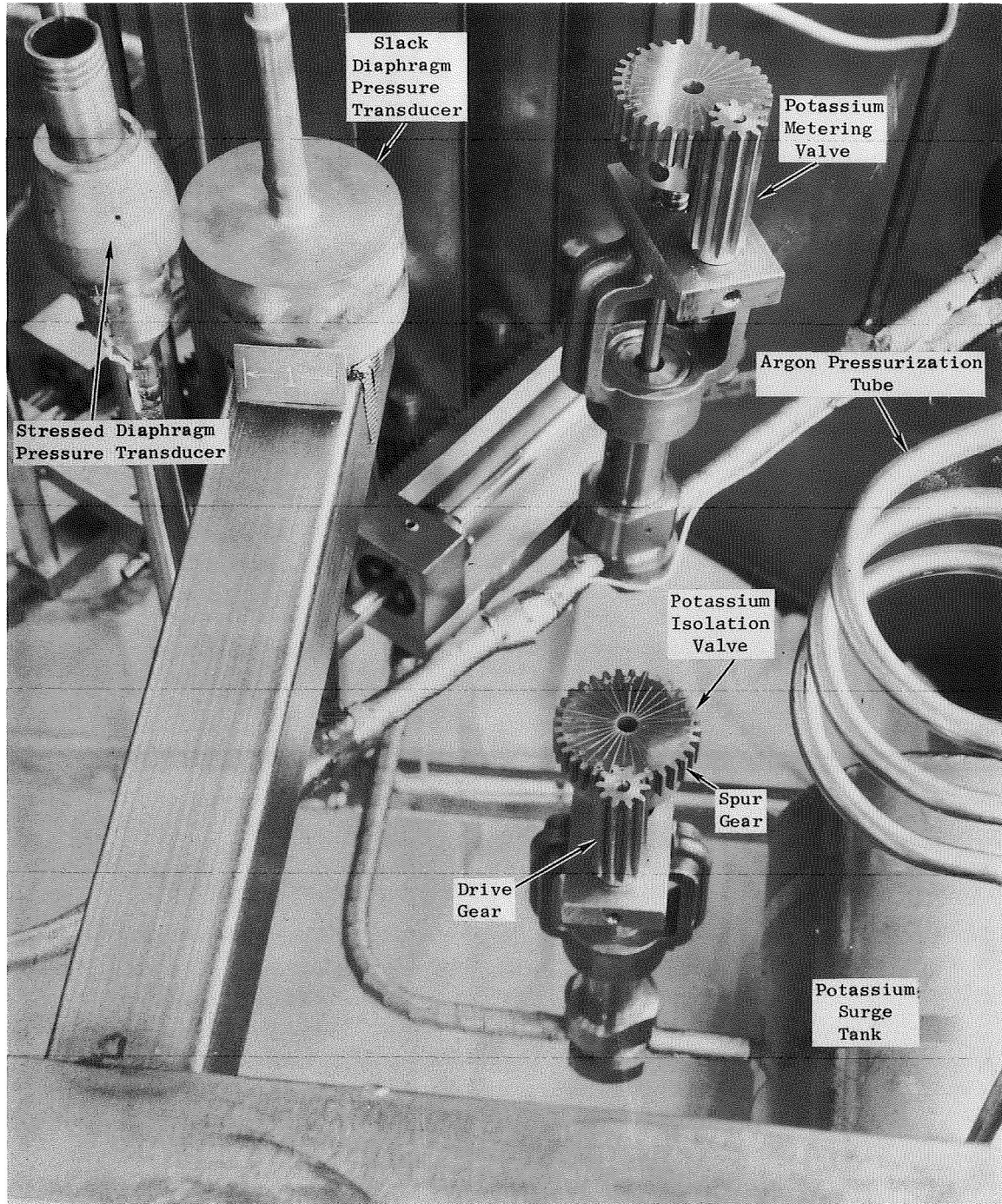


Figure 8. Metering Valve and Isolation Valves - T-111 Rankine System Corrosion Test Loop. (Orig. C68013184)

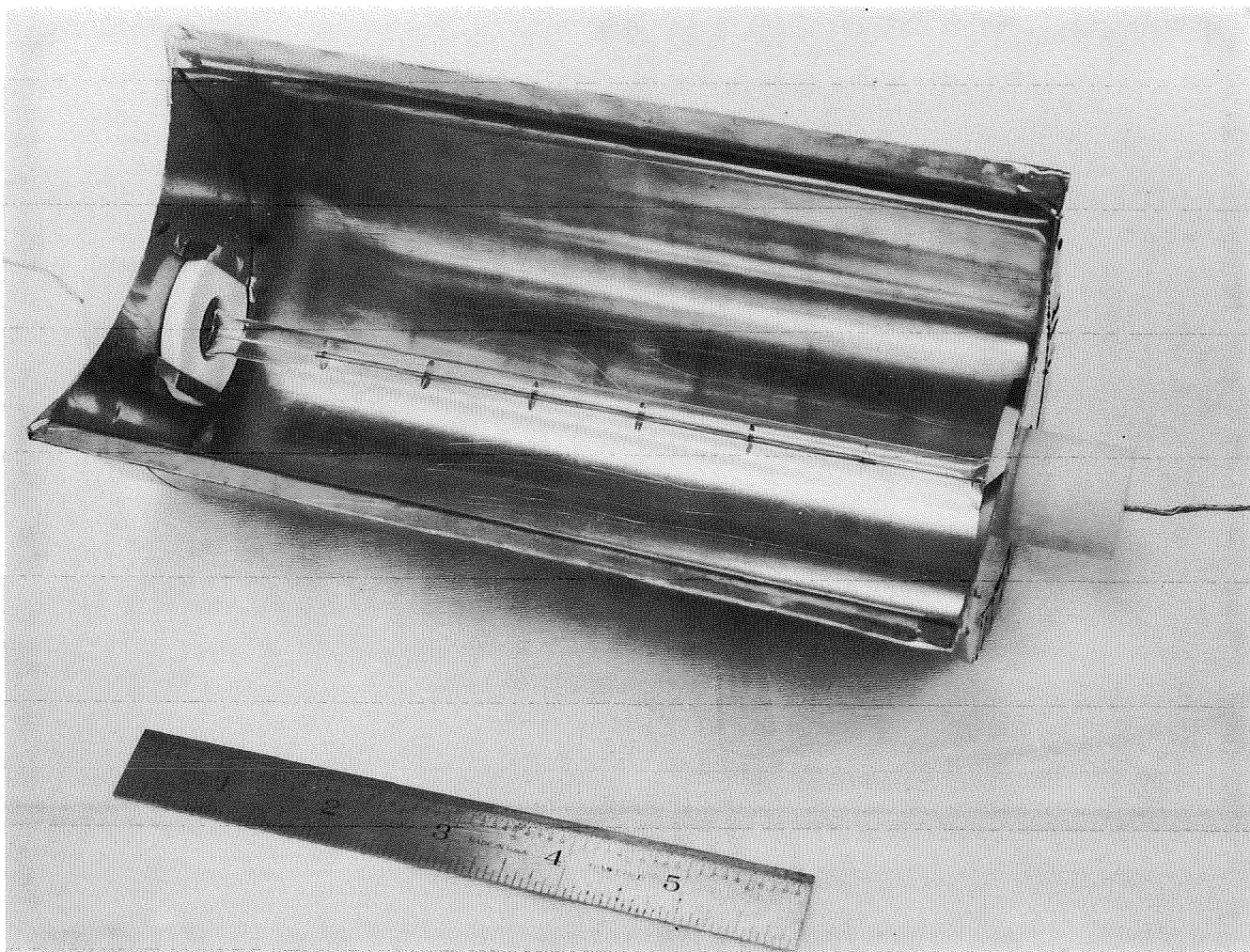


Figure 9. Auxiliary Quartz Lamp Heater Used on the Lithium Surge Tank - T-111 Rankine System Corrosion Test Loop. (Orig. C68013196)

bakeout heaters in melting the lithium in the surge tank after a loop shutdown. The heater will also be used during low power operation to maintain the surge tank above the melting point (m.p. 357°F) of lithium when the bakeout heaters are off.

The adjustable condenser shield assembly, as shown in Figure 10, was the last component to be installed. The shield is actuated by a rack and spur gear drive system and consists of eight movable shield fins constructed of Cb-1Zr. A view of the lower section of the test loop is shown in Figure 11.

2. Pretest Operation of the T-111 Rankine System Corrosion Test Loop

a. Vacuum Chamber Pumpdown. The 48-inch diameter vacuum chamber bell jar was lowered over the T-111 Corrosion Test Loop and bolted to the spool section on February 14, 1968. The vacuum chamber was roughed pumped with the turbomolecular pump to 1×10^{-3} torr before the getter-ion pump was turned on. Although Varian Associates, the manufacturer of the test chamber and pumping system, recommends that the ion-pump be turned on when the vacuum chamber pressure is below 15×10^{-3} torr, it has been found that with the heavy outgassing load from the loop, the ion-pump overheats and will not confine the "glow discharge" at the 15×10^{-3} torr level. The "glow discharge" period is associated with the high pressure operation (5 to 20×10^{-3} torr) when the getter-ion pump voltage is low (approximately 200 volts), the current is high, and the pumping speed is low. During the period of the "glow discharge," electrical discharges having a peak potential of 300 volts have been measured, and the electrical discharge can cause damage to electronic controls that are not grounded or disconnected during this period.

After confinement of the "glow discharge," the pumpdown proceeded slowly, and the pressure remained in the 10^{-4} to 10^{-6} torr range. The pumpdown was accelerated by turning the ion-pump power off and on again after a few minutes to allow the ion-pump to cool. During these periods when the pressure remains constant or even increases, it is believed that gases, especially the inert gases, are being evolved from the hot ion-pump at a higher rate than the ion-pump pumping speed.

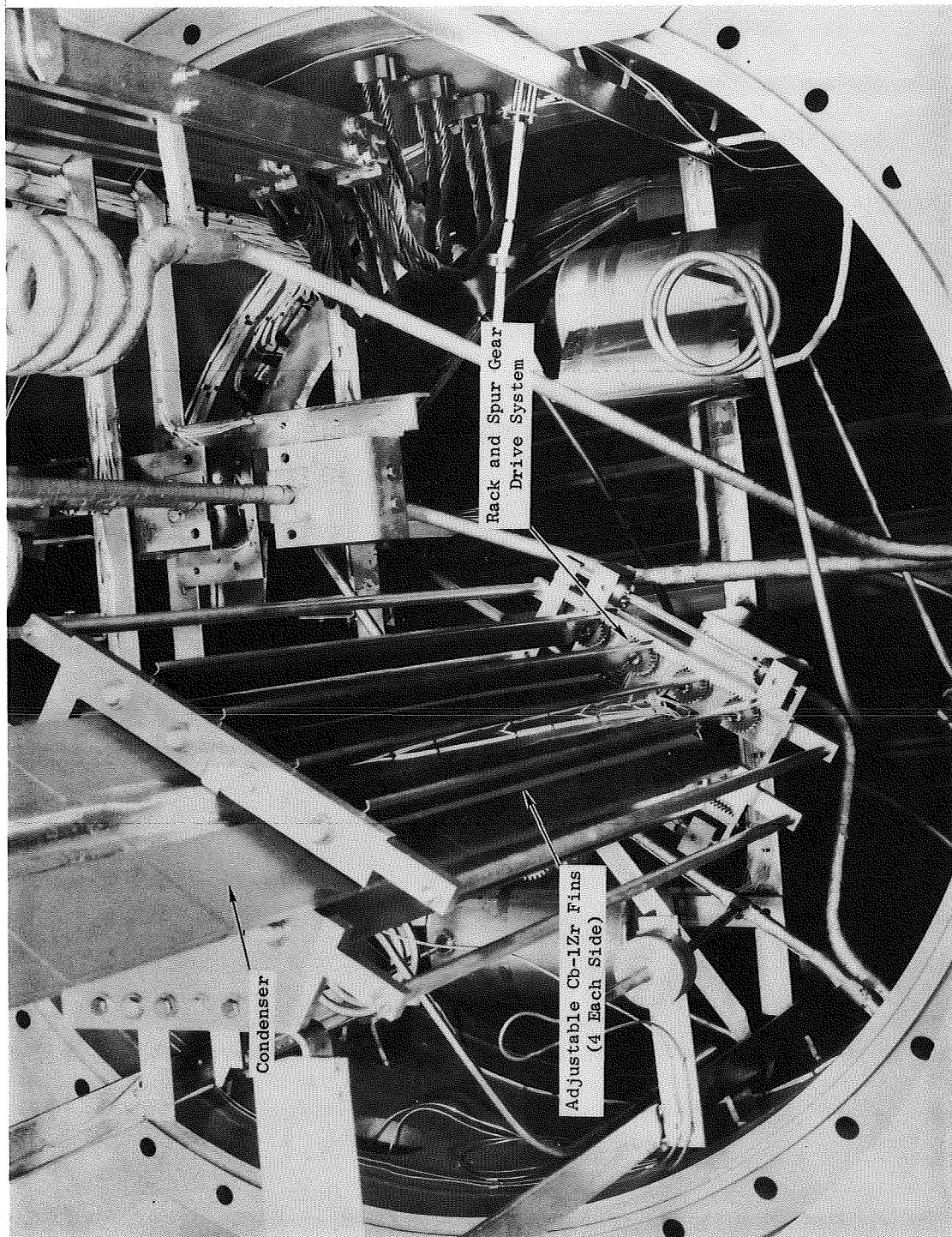


Figure 10. Adjustable Condenser Shield and Drive Mechanism - T-111 Rankine System Corrosion Test Loop. (Orig. C68021350)

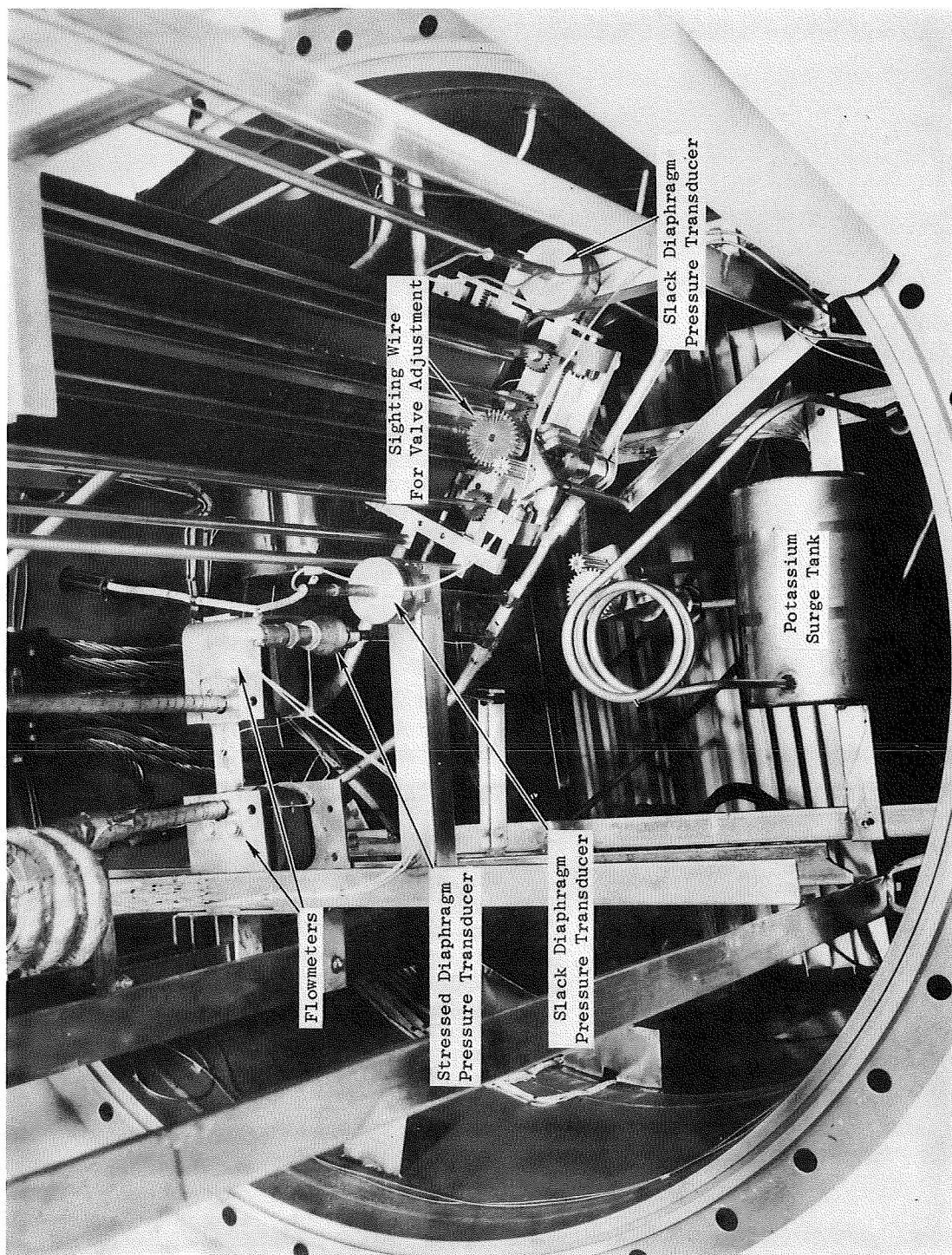


Figure 11. Lower Portion of the T-111 Rankine System Corrosion Test Loop.
(Orig. C68021351)

During the next several days, a number of leak checks were made of all feedthroughs and vacuum chamber flanges. A small leak was found in the main flange which was subsequently sealed by retorquing all the bolts to 175 ft-lbs.

Air cooling ducts were installed on the main flanges since previous tests had shown that the 48-inch diameter Wheeler flange would develop leaks when thermally cycled during the bakeout period. The air-cooled flanges were maintained in the 90°F to 120°F temperature range while the remainder of the vacuum chamber was baked out at 500°F. (A desirable feature of vacuum chambers of this type would be a proportional heater controller, rather than the on-off controller supplied with the system, which would permit a controlled heating and cooling rate for the main flange and also provide better control over the outgassing rate of the loop at the start of the bakeout period.)

The vacuum chamber pressure at the start of the bakeout period was 4.8×10^{-8} torr. The bell jar bakeout heaters were the first to be turned on, followed by the spool section heaters and then the sump heaters as the initial outgassing rate decreased and the getter-ion pump could handle the gas load without overheating. The vacuum chamber pressure increased to the 10^{-6} torr range during this period. After the vacuum chamber pressure returned into the 10^{-7} torr range, the EM pump power was turned on at 10% of the rated power to inductively heat the pump ducts and the insulation cans to accelerate their outgassing rate. A log of the pressure during bakeout is presented in Table I.

b. Final Helium Leak Check of the Test Loop. The T-111 Corrosion Test Loop was helium leak checked after the final assembly weld was made. No indication of a leak was observed at this time. A final leak check of the test loop was completed on February 24, 1968, after the loop was installed in the vacuum chamber and during bakeout prior to filling the loop with alkali metal. The final leak check was made using the partial pressure gas analyzer of the 48-inch diameter vacuum chamber system which is used to measure the residual gas in the vacuum chamber during test operations. The leak check was made by comparing the argon background level in the vacuum chamber with the loop evacuated and with an internal argon pressure of 18 psia. No change in the argon

TABLE I
CHAMBER BAKEOUT
PRESSURE LOG

<u>Date</u>	<u>Hours On Bakeout</u>	<u>Chamber Pressure</u>	<u>Average Loop Temperature</u>	<u>Remarks</u>
2-16	0	4.8×10^{-8}	Room Temperature	Prior to starting bakeout
2-17	24	3.5×10^{-6}	380°F	Bell jar bakeout only
2-18	48	1.2×10^{-6}	400°F	Spool and sump bakeout on
2-19	72	3.0×10^{-7}	400°F	
2-20	96	3.0×10^{-7}	400°F	
2-21	120	2.5×10^{-6}	400°F	Power to EM pumps
2-22	144	3.6×10^{-7}	400°F	
2-23	168	2.3×10^{-7}	400°F	
2-24	192	2.7×10^{-7}	400°F	
2-26	240	1.3×10^{-7}	400°F	
2-27	264	2.5×10^{-7}	400°F	Bake out on partial pressure analyzer
2-28	288	1.0×10^{-7}	400°F	
2-29	312	8.5×10^{-8}	400°F	
3-1	336	8.6×10^{-8}	400°F	
3-2	360	1.2×10^{-7}	400°F	Loop filled with K and Li
3-4	408	1.0×10^{-7}	400°F	
3-5	432	4.0×10^{-7}	525°F	Circulated alkali metals in loop
3-6	456	1.9×10^{-7}	525°F	Start pretest checkout

background level was found during the pressurization of the loop with argon.* The total pressure in the vacuum chamber during the leak check remained at 3×10^{-7} torr.

The filling and flushing of the loop circuits and the sampling of the alkali metals is described in Section B of this report.

c. Checkout and Calibration of the Loop Instrumentation. The only major problem encountered in the equipment checkout was with the 25 KVA voltage stabilizer which was added to the power supply recently to maintain a constant voltage to the test system. During the checkout of the heater power supply, one phase of the three-phase system was overloaded resulting in a blown fuse in the voltage stabilizer. A recheck of the loop power requirements showed that the voltage stabilizer did not have sufficient capacity for the total power input and was disconnected from the circuit. The checkout of the test equipment and loop operation continued without a voltage stabilizer until an available 100 KVA voltage stabilizer was installed.

The calibration of all pressure sensors was repeated with the loop filled with potassium at 500°F and the vacuum chamber on bakeout. The pressure sensors were originally calibrated at room temperature with argon before the loop was filled with alkali metal. All pressure sensors showed good linearity and excellent repeatability over the operating range. The calibration results for the slack diaphragm pressure transducer No. 1 (potassium pump outlet) are shown in Figure 12 and are typical of the results obtained for the other four slack diaphragm transducers.

The calibration of the fast response stressed diaphragm pressure transducer with potassium prior to the test startup is shown in Figure 13. The temperature of the transducer during the liquid metal calibration was 400°F.

*NOTE: Although a leak check across the wall separating the potassium and lithium regions of the tube-in-tube boiler was performed following fabrication of this component, no leak check of this type was performed following installation in the test facility. It is recommended that a check of the later type be considered mandatory for systems of this type in the future.

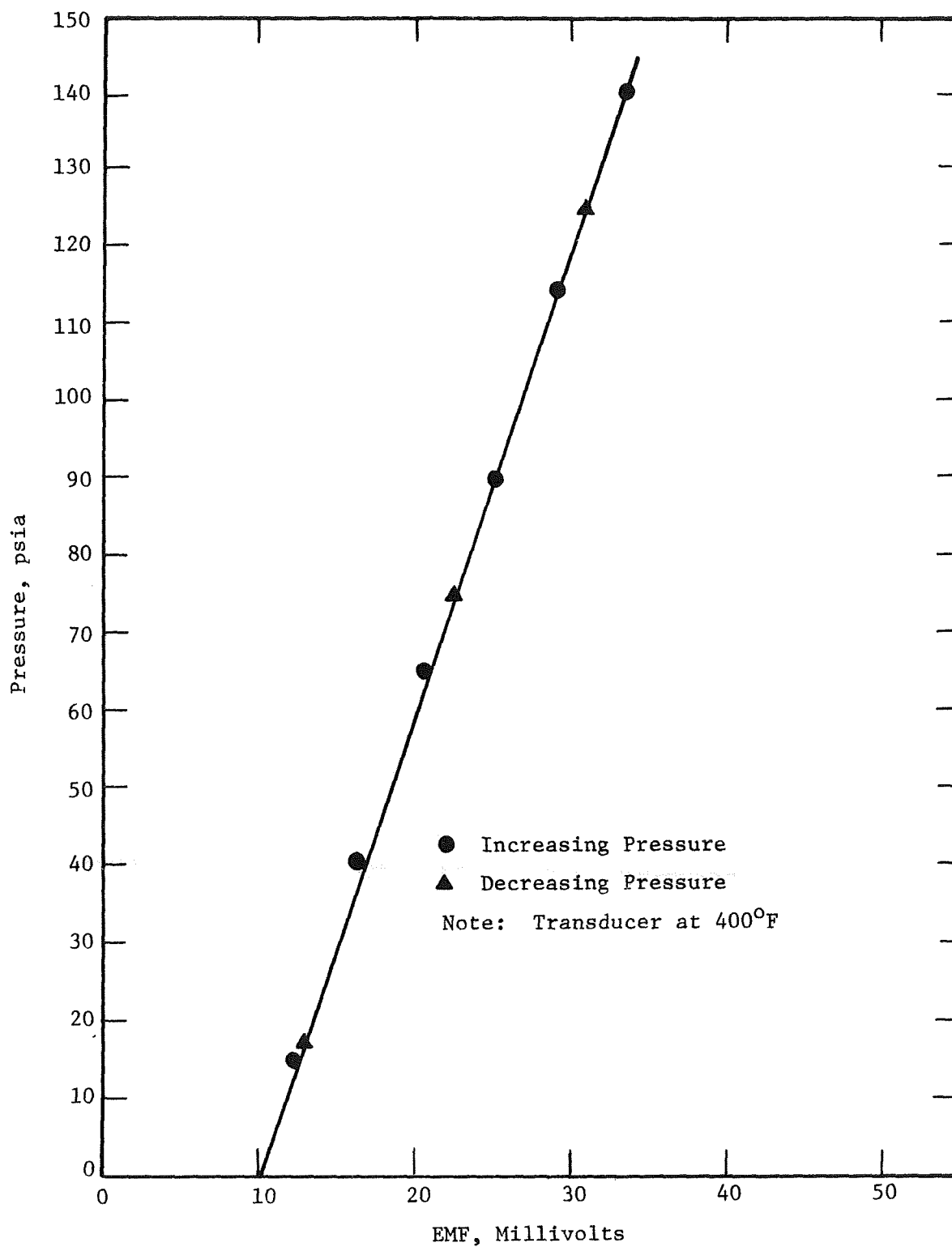


Figure 12. Calibration of Slack Diaphragm Pressure Transducer No. 1 for the T-111 Rankine System Corrosion Test Loop.

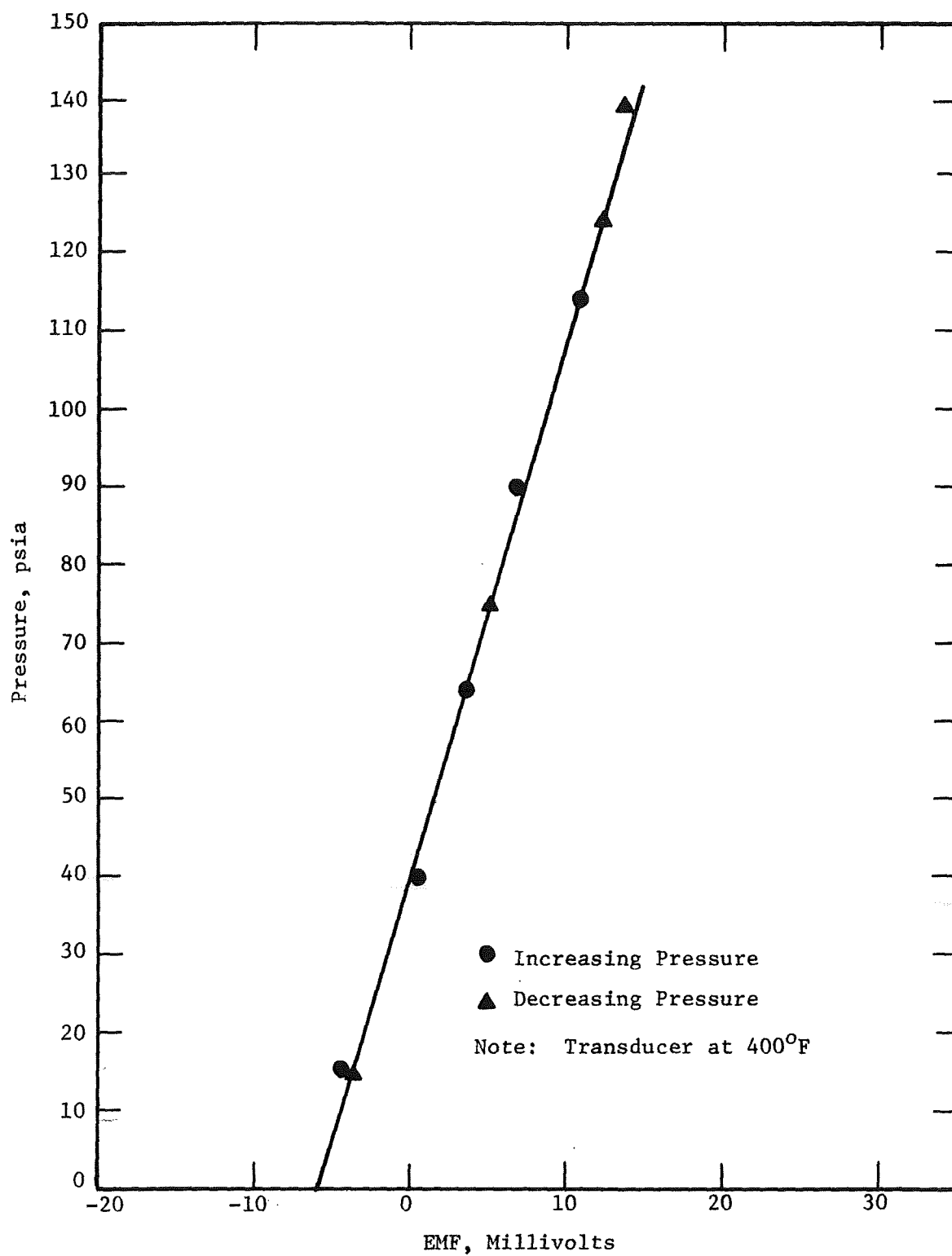


Figure 13. Calibration of Fast Response Stressed Diaphragm Pressure Transducer for the T-111 Rankine System Corrosion Test Loop.

The primary loop thermocouples were checked by operating the lithium circuit at near isothermal conditions and comparing the indicated temperature of each thermocouple with those thermocouples adjacent to it. A near isothermal condition in the primary circuit was achieved by operating at a high lithium flow rate (2 gpm) with the potassium loop empty. The calibration is also useful in computing the heat balance in subsequent loop operation because the temperature drop of the lithium can be equated to the heat loss of the boiler. The temperature distribution as a function of boiler length for the calibration run is shown in Table II. Although higher temperature calibrations are desirable, they were not made at this time because of the high outgassing rate of the loop.

The primary and secondary flowmeters were calibrated by an energy balance across the preheater and heater during all liquid operation. The results of these tests are shown in Figures 14 and 15. The millivolt output as a function of flow rate was considerably lower than that predicted by the theoretical⁽¹⁾ equation for magnetic flowmeters. This discrepancy in flow rates was also observed in the Cb-1Zr Rankine System Corrosion Test Loop flowmeter calibration.⁽²⁾ The T-111 Corrosion Loop flow tubes are 0.25-inch ID x 0.065-inch wall thickness with a ratio of outer diameter to inner diameter of 1.5:1, which is considerably larger than the diameter ratio (1.12:1) used by Affel, et al. in the referenced report.⁽¹⁾

After completion of the calibration tests, the loop safety circuits were checked and set according to the procedures outlined in the test plan.⁽³⁾

-
- (1) Affel, R. G., Burger, G. H., and Pearce, C. L., Calibration and Testing of 2- and 3 1/2-Inch Magnetic Flowmeters for High-Temperature NaK Service, Oak Ridge National Laboratory, ORNL-2793, p. 16.
- (2) Potassium Corrosion Test Loop Development Program, Quarterly Progress Report No. 8 for Period Ending July 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54843, November 23, 1965, p. 25.
- (3) Advanced Refractory Alloy Corrosion Loop Program, Test Plan for T-111 Rankine System Corrosion Test Loop, NASA Contract NAS 3-6474.

TABLE II

RESULTS OF CALIBRATION^(a) OF W-3Re/W-25Re THERMOCOUPLES IN THE BOILER
SECTION OF THE T-111 RANKINE SYSTEM CORROSION TEST LOOP

Thermocouple No.	Temperature °F	Location Inches From Boiler Exit ^(b)
B1	905	0
B2	903	2
B3	902	4
B4	903	6
B5	916	9
B6	914	12
B7	914	15
B8	914	18
B9	910	30
B10	908	43
B11	908	55
B12	908	68
B13	908	80
B14	908	93
B15	908	105
B16	908	118
B17	914	130
B18	914	143
B19	914	155
B20	911	168
B21	909	180
B22	909	193
B23	908	205
B24	908	218

(a) Calibration test conducted with a lithium flow of 2.0 gpm in the primary circuit and no potassium in the secondary circuit.

(b) Lithium exit, potassium inlet.

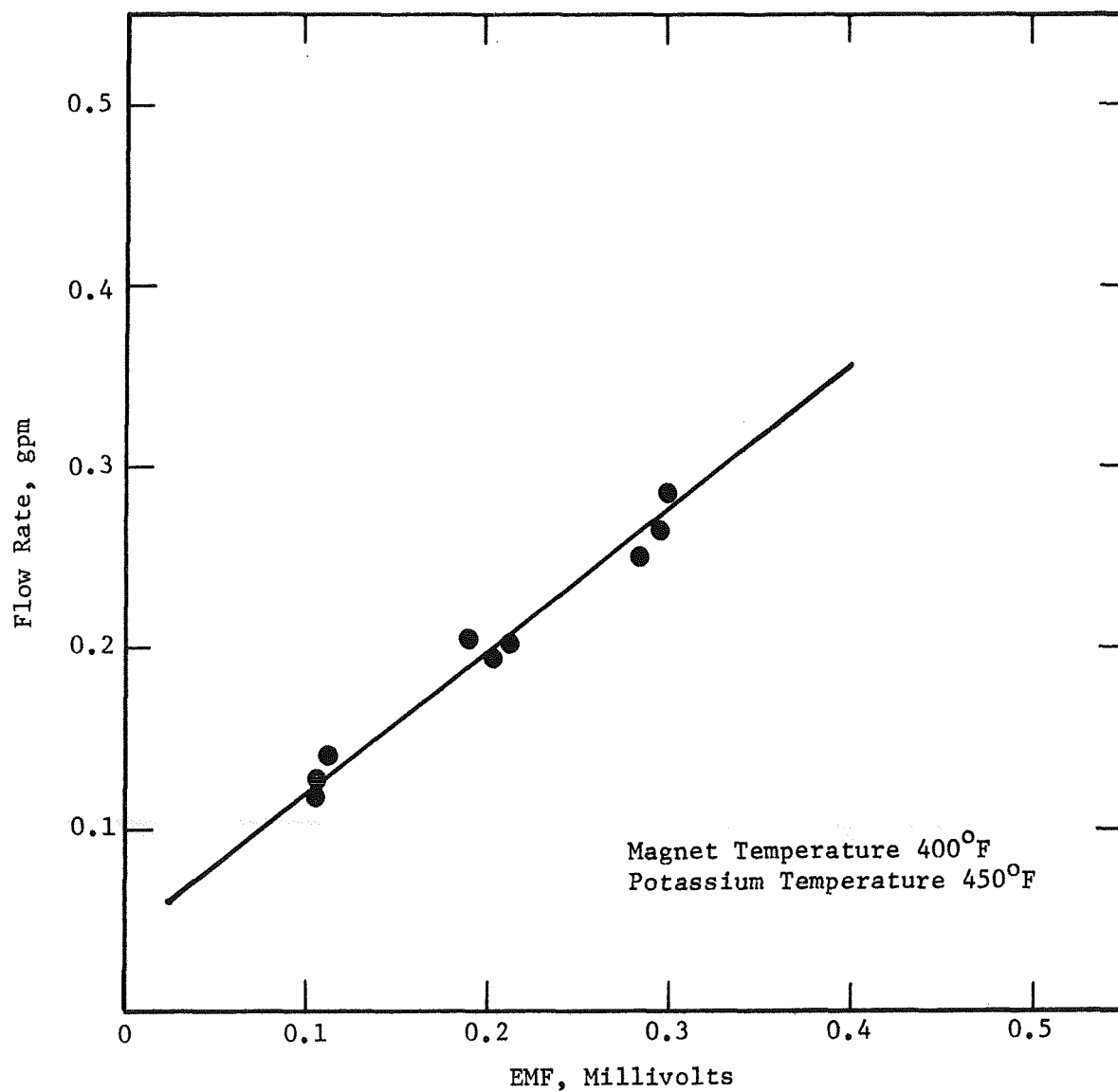


Figure 14. Potassium Flowmeter Calibration - T-111 Rankine System Corrosion Test Loop.

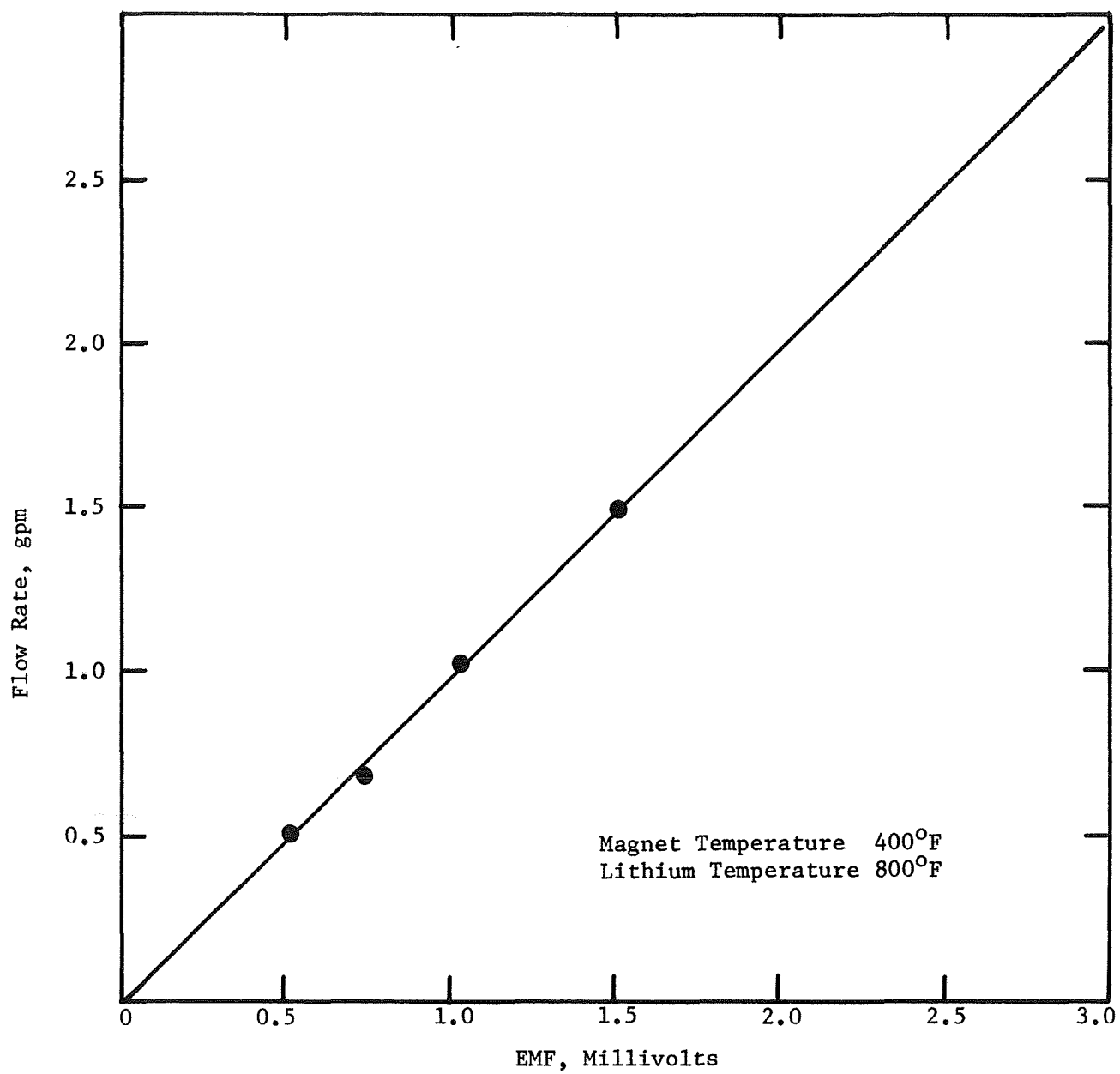


Figure 15. Lithium Flowmeter Calibration - T-111 Rankine System Corrosion Test Loop.

3. Operation of the T-111 Rankine System Corrosion Test Loop

The boiling and condensing operation of the T-111 Corrosion Test Loop was started on March 11, 1968, after completion of the checkout of the loop control and safety circuits. The test loop had completed one week of outgassing with all liquid operation and was at the test conditions listed below at the start of the boiler and condensing operation:

Vacuum Chamber Pressure	4×10^{-7} torr
Lithium Flow Rate	2 gpm
Potassium Flow Rate	0.2 gpm
Lithium Temperature	1180°F
Potassium Temperature	1140°F

The potassium loop was dumped by evacuating the surge tank and gravity draining the potassium from the loop into the surge tank. The metering valve was adjusted to 3/16 open which is the position determined from the valve calibration test as the setting required for a 10 to 25 psia pressure drop across the valve at the design operating conditions.

The potassium surge tank was pressurized with argon to 2.0 psia, forcing the liquid potassium out of the surge tank into the loop. This pressure is sufficient to partially fill the loop to the inlet to the boiler and approximately 2/3 of the condenser. The boiling operation was started at 0800 March 11, 1968, by turning on the potassium EM pump at low power and forcing liquid potassium into the boiler. The potassium flow rate and the lithium temperature were slowly increased and allowed to stabilize at a potassium vapor temperature of 1250°F and a lithium temperature of 1366°F. The vacuum chamber pressure increased to the 10^{-6} torr range as the loop temperature reached a new high and further power increases were delayed until the vacuum chamber pressure decreased into the 10^{-7} torr range.

During the next several hours, the loop temperature and potassium flow rate were slowly increased until at 1530 March 11, 1968, the potassium flow rate slowly decreased to zero flow. At this time all pressure gauges in the potassium loop, shown in Figure 2, were at the same pressure indicating the flow blockage between the condenser and the

pump inlet. The potassium flow direction was reversed and the pressure gauges No. 2, 4, 5, and 6 then read the same pressure, but pressure No. 1 read zero indicating the flow blockage was then at the metering valve. The metering valve was then opened from 3/16 open to full open and all pressure gauges responded and a high potassium flow rate was established.

The potassium surge tank was immediately evacuated in an attempt to dump the loop and remove the plugging material from the loop to the surge tank. The loop would not dump indicating that a flow blockage was present between the surge tank and the loop. The potassium surge tank was pressurized in an attempt to dislodge the plugging material however, no immediate response on the loop pressure gauges was observed. After a few minutes, the loop pressure slowly increased to 80 psia (the pressure in the surge tank). The surge tank was again evacuated but the loop would not dump, and the pressure in the loop remained at 80 psia. The vacuum chamber bakeout system was turned on at 1900 hours since the loop was now operating at a lower power level and the alkali metal could freeze if additional heat was not supplied to the loop. At 2015 hours communication between the surge tank and the loop was established suddenly and the loop and surge tank pressures equalized to 18 psia. The potassium EM pump was turned on, and potassium could be circulated at a high flow rate. The plug between the loop and the potassium surge tank could have been frozen potassium in a cooler location; however, the temperatures in this general region were significantly above the 147°F melting point of potassium.

At 0800 March 12, 1968, a second attempt to reach the design operating conditions was made. The loop power was slowly increased, but extremely large flow fluctuations made the loop difficult to control. The possibility that additional plugging material remained in the loop after the previous dump was considered as a potential source of the instabilities and, consequently, at 1630 hours the potassium circuit was dumped and the loop refilled after holding the loop charge of potassium in the surge tank for 10 minutes. Loop operation continued and the lithium temperature was increased to 1770°F, but at 1900 hours large pressure and flow fluctuations again prevented reaching design operating conditions.

At 0800 March 13, 1968, a third attempt was made to reach the design operating conditions. The potassium circuit was dumped and again held in the surge tank to allow time for settling of particulate material before refilling the loop. The loop power was slowly increased with excellent loop stability until the following conditions were reached: lithium temperature, 2030°F; potassium boiling temperature, 1750°F; boiler outlet potassium vapor temperature, 2000°F. The vacuum chamber pressure at this time was 9.5×10^{-7} , and additional power increases were delayed to await a decrease in the vacuum chamber pressure. The test operation continued at these conditions until 1230 March 14, 1968.

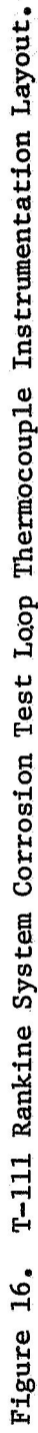
The loop power was again increased in an attempt to reach the design operating conditions. The power level was increased to the maximum available from the 20 KVA heater power supply, but the design conditions could not be reached. The test data was recorded at this time and is shown in Figure 16. The test was subsequently shut down to add more thermal insulation to the loop to reduce the heat losses in the lithium circuit and thereby obtain the higher temperatures required to meet the design conditions.

The 48-inch diameter vacuum chamber was opened on March 15, 1968, to add additional thermal insulation to the lithium heater to reduce radiation losses. The heat shields shown in Figure 17 consist of a 0.005-inch thick tantalum inner shield with three 0.002-inch thick tantalum shields separated by 0.020-inch tantalum wire spacers. The heat shields were assembled in place by spot welding the individual shields together to form an integral assembly and then the ends were reinforced with a 0.060-inch thick stiffening ring. The thermal shield assemblies were positioned around the heater coils by bolting the stiffening rings to the grounded heater bus bar. The clearance between the inner shield and the heater coil was approximately 0.5-inch. The addition of the four shields is designed to reduce the heat loss from the heater coils by approximately 75%.

An additional layer of 0.002-inch dimpled foil was also added to the tubing between the lithium heater exit and the inlet to the boiler.

The 25 KVA voltage stabilizer on the loop power supply was

Chamber Pressure: 1.1×10^{-6} Torr
Heater: 40.5A/420V/17.1KW
Preheater: 35A/93V/3.2KW
Primary Flow: 2.75 MV/2.75 GPM
Secondary Flow: 0.15 MV/0.16 GPM
Boiling Temperature: 1910°F
Superheat: > 200°F



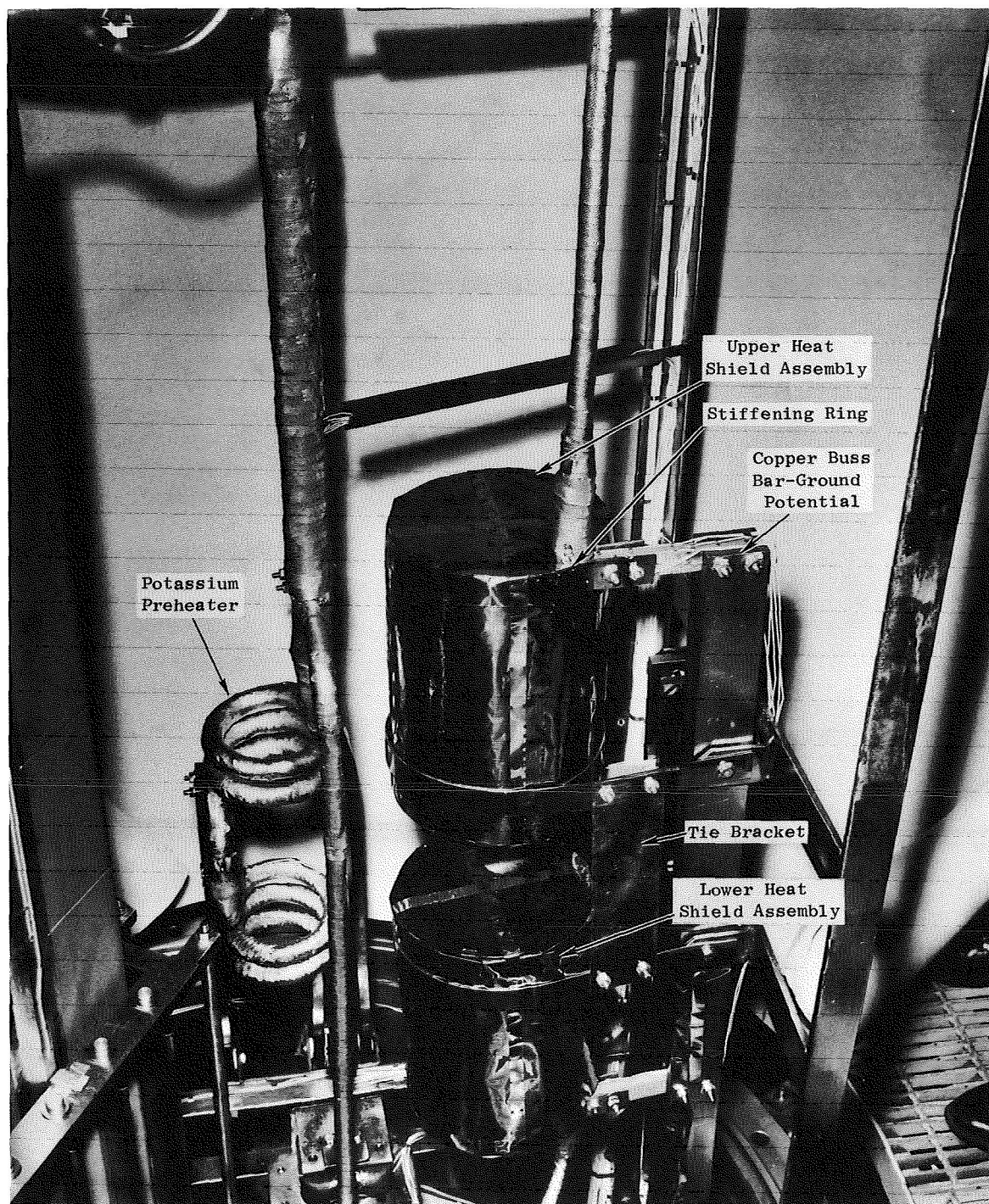


Figure 17. Rankine System Corrosion Test Loop Lithium Heater Thermal Insulation Cans. (Orig. C68032928)

replaced at this time with a 100 KVA unit that will be capable of handling the electrical power input required to meet the design test operating conditions.

The bell jar of the 48-inch vacuum chamber was lowered and bolted to the spool section flange on March 3, 1968. The vacuum chamber was rough pumped overnight with the turbomolecular pump to 1×10^{-3} torr before the ion-pump was started. The bakeout heaters were turned on when the vacuum chamber pressure was in the 10^{-7} torr range. All liquid loop operation with a maximum temperature of 1100°F was continued for several days to outgas the thermal shields that were added to the lithium heater.

At 0800 March 25, 1968, boiling and condensing operations were resumed. The potassium loop was dumped into the surge tank and the potassium allowed to remain in the surge tank under vacuum for 10 minutes before the loop was refilled by pressurizing the surge tank with argon. The power input to the loop was slowly increased as the vacuum chamber pressure improved. At 1510 hours, the maximum power input to the heater had been applied but was not sufficient to bring the loop to its design operating condition. An attempt to maintain the loop at the maximum power level and obtain steady state test data was not successful because of the changing potassium flow rate. The potassium flow rate steadily decreased and the pressure drop across the valve increased indicating that the valve was plugging with particulate matter. The metering valve was opened from 3/16 open (the original setting) to 1/4 open with an instantaneous increase in the potassium flow rate and decrease in the pressure drop across the metering valve as shown in Figure 18. The flow rate again decreased as the valve was opened in steps up to full open and the plugging noted above occurred after holding for a few minutes at each new valve opening. When this occurred with the valve fully open, the heater and preheater power were quickly reduced to a safe level to avoid overtemperaturing of these components. The vacuum chamber bakeout heaters were turned on to maintain the loop above the melting point of the alkali metals.

The potassium surge tank was then evacuated to dump the potassium into the surge tank in an attempt to remove the plugging material from the loop. The potassium was allowed to settle in the surge tank for

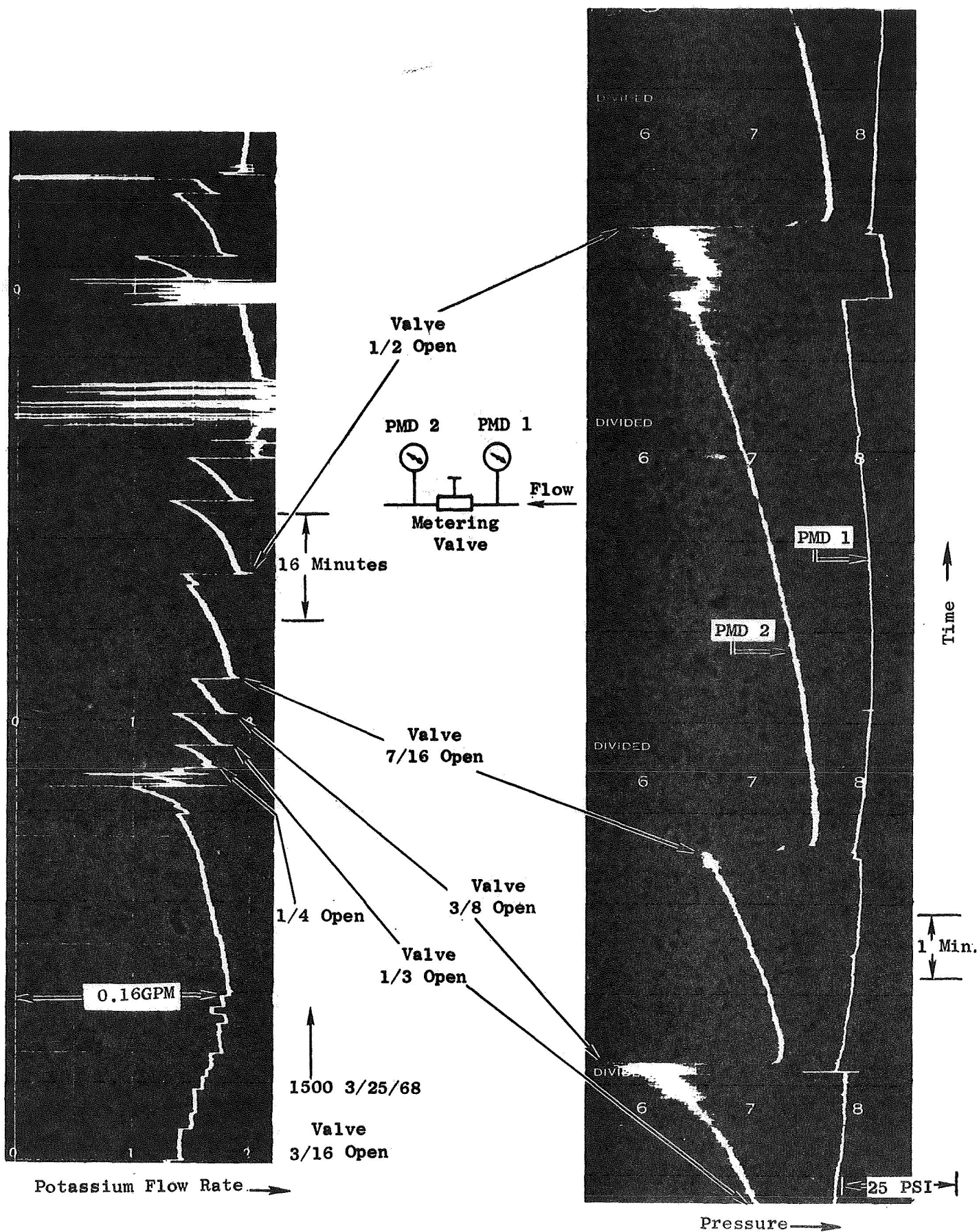


Figure 18. The Effect of Plugging the Metering Valve on Potassium Flow Rate and Pressure Drop Across the Metering Valve During Operation of the T-111 Rankine System Corrosion Test Loop.

approximately 10 minutes before the loop was refilled by pressurizing the surge tank. The potassium pump power leads were changed for reverse flow and a high potassium flow could be maintained, indicating the loop had unplugged. The loop was again dumped, refilled with potassium and circulated in the reverse direction as before. The dumping procedure was repeated for the third time and again the normal flow rate was initially established but then it slowly decreased to zero. The EM pump was rewired for forward flow but potassium circulation could not be established. During this period, the metering valve was operated repeatedly in an attempt to unplug the loop. The valve operation became progressively worse requiring a higher torque with each operation until the valve could no longer be operated in either direction. The valve remained in the open position after the final adjustment was made.*

The potassium and lithium were dumped into the surge tanks and plans were made to sample the potassium inventory in an attempt to identify the plugging material before the test operation could continue. The draining and sampling of the loop circuits are described in the following section (B.2.) of this report. A summary of loop operations on March 25, 1968, is presented in Table III.

B. ALKALI METAL PURIFICATION AND CONTROL

1. Pretest Alkali Metal Quality Control

During this quarter, the lithium and potassium purification and transfer systems were attached to the loop fill lines in order to fill the primary and secondary circuits. All liquid metal transfer lines and gas pressurization lines outside of the purification and transfer system ovens were traced with heating tapes and heated to 250°F to 700°F under vacuum for ten days to outgas the lines. Both systems were helium leak checked at temperature according to SPPS Specification 03-0013-00-B, "Mass Spectrometer Leak Detection Using Helium," and no leaks were found in excess of 5×10^{-10} std. cc of air per second. The pressure rise rates measured with the lines at temperature were 6×10^{-4} torr-liters per minute for the potassium system and 2×10^{-4} torr-liters per minute for the lithium system prior to transfer of the alkali metals. The test plan specified that the pressure rise rate shall be less than 10^{-3} torr-liters per minute.

* When the bell jar was removed from the test facility inspection of the valves indicated binding of the Mo-TZM pinion gear shaft on the tungsten carbide sleeve bearing resulted in the valve operation difficulties.

TABLE III

T-111 RANKINE SYSTEM CORROSION TEST LOOP TEST LOG ON 3-25-68

0030	Vacuum Chamber on Bakeout - Loop on Low Power With All Liquid Flow Since 3-22-68 - Test Data Recorded and Partial Pressure Gas Scan #45 Made T Max = 1100°F
0300	Increased Heater Power to 22A 170V T Max 1150°F
0530	Increased Heater Power to 25A 200V T Max 1325°F
0600	T Max 1450°F - Chamber Pressure 4.5×10^{-7} Torr - Leak Checked All Flanges - No Leak Indications
0800	Hot Dumped Potassium Loop and Partially Refilled Loop With 2 psia Pressure
0825	Closed Metering Valve to 3/16 Open (2.25 Div)
0900	Chamber Pressure 7.5×10^{-7} Torr - Completed Leak Check - No Leaks Indicated
0927	Start Boiling Operation by Turning on Potassium Pump - Increased Power Input and Potassium Flow Rate as Vacuum Pressure Permitted
1000	Heater Power 35.5A 300V - Bakeout Turned Off - Turbo Molecular Pumped Valved Out of System
1010	Continued Increasing Power and Potassium Flow Rate
1130	Complete Set of Data Loop Stabilized to Improve Vacuum Before Increasing Loop Temperature 1960°F
1145	Vacuum Chamber Cooling Water Turned On
1200	Partial Pressure Gas Analysis No. 48
1300	Start of Minor Power and Potassium Flow Adjustments to Reach Design Operating Conditions
1510	Power Input to Heater 40A and 440V - Attempt to Attain Thermal Equilibrium to Record Test Data
1525	Potassium Flow Decreasing - Open Valve to 3.75 Divisions
1530	Flow Decreasing - Open Valve to 4.0 Divisions
1533	Flow Decreasing - Open Valve to 4.75 Divisions
1545	Flow Decreasing - Open Valve to 5.5 Divisions

TABLE III (Cont'd)

1555	Flow Decreasing - Open Valve to 6.0 Divisions
1600	Flow Decreasing - Valve Fully Open
1620	Flow Decreasing - Valve Half Open
1700	Flow Decreasing - Valve Full Open
1830	Potassium Loop Dumped - Surge Tank Temperature Increased From 460°F to 560°F
1845	Potassium Flow Reversed - High Flow Rate
1845	Potassium Flow Returned to Normal Direction - High Flow Rate
1847	Dump Potassium Loop - No Increase in Surge Tank Temperature
1855	Filled Loop and Flowed at High Rate
1900	Dumped Potassium - Surge Tank Temperature Increased From 502°F to 533°F
1905	Re-filled Loop and Reversed Flow - No Flow Rate PMD 1 and PMD 2 Show No Pressure
1910	Flow Oscillations Indicating Boiling in the Preheater Section of the Loop
1915	Dumped Potassium Loop - Surge Tank Temperature Did Not Change
1917	Dumped Lithium Loop - Surge Tank Pressure Increased From 4.7 mv to 8.5 mv
1918	All Power on Loop Off - Chamber on Bakeout

The transfer systems were filled with liquid metal which was subsequently dumped into the disposal tank to flush the systems. The systems were refilled and a sample removed from each charge pot for chemical analysis. Oxygen and nitrogen analyses indicated that these impurities were below the specified limits of 20 ppm nitrogen and 150 ppm oxygen, and the lithium in the charge pot was then transferred to the evacuated lithium circuit surge tank. The MSAR^(a) inductive liquid level probe in the charge pot was used to assure that the complete charge had been transferred. The argon pressure in the gas line was measured before and after the transfer; i.e., both before and after the argon had expanded into the charge pot. The lithium surge tank was then pressurized with argon, forcing the lithium into the loop. The lithium was circulated for 1 hour at 500°F and then dumped back into the surge tank. A sample of lithium was then removed from the surge tank for chemical analysis.

The oxygen concentration in the potassium sample taken after flushing the transfer line was found to be below the specified limit of 20 ppm. Since the potassium charge pot is not equipped with a level probe, the quantity transferred was determined by pressurizing the charge pot with argon to the same pressure employed during the lithium filling operation. The flow of potassium was stopped when the pressure had dropped to the same value noted after the lithium was transferred. Since the volumes of the lithium and potassium charge pots and gas lines are the same, this technique was believed adequate. Subsequent investigation, which will be described in the next progress report, has indicated that only about 60% of the desired charge was transferred to the potassium surge tank.

After circulating the potassium for 1 hour at 500°F it was returned to the surge tank and sampled. The lithium and potassium loop circuits were then pressurized for low temperature, all liquid, operation to check out the loop controls and safety circuits and to calibrate the loop instrumentation. During this period of low temperature operation, chemical analyses were performed on the alkali metals to insure that they were still within the operating specification and the loop could be operated

(a) Mine Safety Appliance Research, Callery, Pennsylvania.

at the design conditions. The complete analytical data are presented in Table IV.

The analytical chemical criteria for filling and operating the loop circuits are that the potassium contain no greater concentration of oxygen than 20 ppm and that the nitrogen and oxygen concentrations in the lithium be no greater than 20 ppm and 150 ppm respectively. As shown in Table IV, the oxygen concentration in the potassium was within limits, as analyzed by the mercury amalgamation method, in samples taken before filling the transfer system (No. 1405) after filling the charge pot and transfer system (No. 1487A) and after flushing the loop (No. 1493A). The high oxygen concentration in potassium indicated by Gulf General Atomic fast neutron activation analysis (No. 1405) is considered to be in error and was discounted because it has been impossible in recent months to obtain agreement with the amalgamation method on this and other potassium samples of known low oxygen concentration.

It should be pointed out that the decision to proceed in the filling operation is based on the chemical analytical criteria for oxygen and nitrogen given above, and that the analyses for metallic impurities are not completed until several days after a particular transfer has been made. The high values obtained for such elements as iron, chromium and nickel for samples 1487A and 1493A were originally attributed to inadvertant contamination during sampling subsample preparation or analysis since no reasonable source for these stainless steel components could be defined. However, additional specimens were submitted for reanalysis by both GE and NSL^(a). The results indicated for Samples 1487B and 1493B would lead one to believe that inadvertant contamination of the "A" samples had indeed occurred, if it were not for the fact that particulate material has been found in the potassium subsequent to the shutdown caused by the plugging of the metering valve.

The analytical results shown for lithium Samples 1355, 1488 and 1492 indicated that the nitrogen concentration was within the specified limit (20 ppm) prior to each transfer. The results for oxygen obtained by thermal neutron, fast neutron and distillation techniques indicate that the oxygen concentration was well below the specified 150 ppm.

^(a) National Spectrographic Laboratory, Cleveland, Ohio.

Sample Number and Date	Identity	Analytical Lab. ^a									Remarks
			Sn	Sr	Ta	Ti	V	W	Zn	Zr	
292 1-4-66	Lithium: As received	GE	<25			<5	<25			<5	
		GGA (FN) ^b									
1588 3-25-68	Lithium: As received. Resampled	GE (D)									
293 1-4-66	Lithium: After filtering at 400°F through a 5-micron filter into the hot trap	GE	<25			<5	<25			<5	
		GGA (FN)									
309 4-4-66	Lithium: After hot trapping at 1500°F for 126 hours. Sampled at 500°F	GE	<25			<5	<5			<5	
		GGA (FN)									
1020 4-20-67	Lithium: Residue dissolved out of 15-micron filter which plugged while trying to return lithium from receiver to hot trap after overflowing still pot into receiver. Metals reported as ppm in the residue	GE	ND	25		10	5			ND	
1031 5-4-67	Lithium: After distilling 15 lbs in 3 5-lb batches. The first two batches were returned to the hot trap and the third batch was sampled at 365°F from the receiver. No filter present at this time	GE	<25	<5		<25	<25			<25	
		GGA (FN)									
1064 6-1-67	Lithium: Second sample from 5-lb batch (See Sample 1031). Sent to AEC	GE									
		GGA (FN)									
1313 10-26-67	Lithium: Third sample from 5-lb batch (See Samples 1031 & 1064). Sampled at 375°F	GE									
1355 12-5-67	Lithium: Sampled at 425°F from still receiver after distilling for the T-111 loop fill	GE	<5	<5		<5	<50			<50	
		GGA (FN) ^b									
		ORNL (TN)									
		ORNL (FN)									
		NASA (D)									
1488 3-1-68	Lithium: From charge pot and transfer system. Taken at 400°F	GE (D)	<25	5		<25	<25			<25	
		NSL			<250						
1492 3-4-68	Lithium: After loop flush. Taken at 400°F	GE (D)	<50	5		<5	<25			<25	
		NSL			<250						
		ORNL (FN)									

Sample Number and Date	Identity		Analytical ^a Lab.	ppm							Results
				O	Ta	Ti	V	W	Zn	Zr	
1578	Lithium:	Drained from still pot after	GE (D)	96		<25	<25			<25	Potassium determined by flame photometry
3-20-68		distilling for T-111 loop fill		110							
1683	Lithium:	From transfer line used between	GE								
4-22-68		valve FF on the transfer system and valve KK on the loop for filling and flushing the loop initially									
190	Potassium:	As received	GE (A) ^b	4		<2	<2			<10	Lithium determined by flame photometry
4-21-65				6							
1352	Potassium:	From still receiver after	GE (A)	2; 1		<2	<20			<20	
12-1-67		distilling for T-111 loop fill									
1405	Potassium:	Second sample from still receiver.	GE (A)	5		<2	<10			<2	Lithium determined by flame photometry
1-5-68		Taken at 270°F. Taken especially for comparison of amalgamation and fast neutron activation results for oxygen	GGA (FN)	257							
				212							
				193							
1487 A	Potassium:	From charge pot and transfer lines.	GE (A)	17		<10	<30			<30	Lithium determined by flame photometry
2-29-68		Taken at 250°F		20							
1487 B	Potassium:	Four inches of potassium extruded	GE			<2	<20			<10	
2-29-68		from 1/2-inch x 20-mil wall sample tube and converted to the chloride to try to pick up high stainless steel concentrations									
1493 A	Potassium:	After loop flush. Taken at 400°F	GE (A)	14		<10	<30			<30	Lithium determined by emission spectrography Lithium determined by flame photometry
3-4-68				14							
1493 B	Potassium:	Subsample preparation repeated for	GE			<10	<10	<100	<100	<10	
3-4-68		additional spectrographic analyses	NSL			<20	<20				
1622	Potassium:	From the transfer line used between	GE			<2	<20			<10	Lithium determined by flame photometry. A few black particles found at bottom of tube
3-29-68		valve FF on the transfer system and valve KK on the loop for initial filling and flushing operation									
1668 A	Potassium:	From bottom third of the sample tube	GE (A) ^b	7, 7		<2	<20			<10	
4-10-68		during drain of secondary (See Fig. 1)									
1668 B	Potassium:	From a portion of the heavy walled	GE								Lithium by flame photometry. Black particles found during preparation of chlorides
4-10-68		tubing used as the transfer line between valve KK and the still pot (See Fig. 1)									
1668 C	Black particles found during preparation of		GE		ND	Mi		ND	T	T	
4-10-68	1668 B										
1669 A	Black, particulate, residue which remained in still	NSL									Fe = 15 - 25%, Cr = 25 - 50%, Mn = 25 - 50%, Ni = 5 - 15%, Si = 5 - 15%, Ti = 0.1 - 0.5%, Al = 0.1 - 0.5%, V = 0.1 - 0.5%, Ta < 0.05% X-ray diffraction indicated β-LiFeO ₂ and δ-LiFe ₅ O ₈ initially. After standing in air, saw only Li and Fe X-ray diffraction indicated Li, Li ₂ O, LiOH and δ-Fe. No Cr, Mn or Ti seen
4-11-68	pot (Fig. 1) after distillation - swept into vial with a brush. Compare with sample 1668 C. Specimen for microscopic examination is still available										
1669 B 1	Sample for x-ray diffraction. Capillary	GE									
4-11-68	loaded under argon										
1669 B 2	Sample for x-ray diffraction. Capillary	GE									Still available
4-11-68	loaded in air										
1670	Black, particulate, residue from still pot										
4-11-68	(Same as sample 1669 A), but sucked into Erlenmeyer flask with reduced pressure										
1671	Residual metal (Li) scraped from still pot										Still available
4-11-68	(Fig. 1), and placed in sealed vial										
1672	Residual metal removed from still pot (like	GE									
4-11-68	sample 1671) by dissolving in water										
1682	Potassium:	From distillation receiver after	GE								Lithium and potassium determined by flame photometry
4-18-68		the distillation (See Fig. 1). Taken at 275°F during disposal									
Notes: a. Analytical Laboratories are designated as follows: GE - General Electric Company, Evendale, Ohio GGA - Gulf General Atomic, San Diego, California ORNL - Oak Ridge National Laboratory, Oak Ridge, NASA - National Aeronautics and Space Administra- NSL - National Spectrographic Laboratory, Clevel-											
b. Initials in parentheses designate the method used: A - Amalgamation FN - Fast Neutron Activation TN - Thermal Neutron Activation D - Distillation											

The oxygen results from Gulf General Atomic for the lithium samples are considered inaccurate. This decision was reached based on the results of a large number of analyses obtained by other laboratories using methods considered to be reasonably accurate. The results obtained for metallic concentrations are puzzling because it is almost impossible to envision a source of such impurities subsequent to distillation especially since the lithium is filtered at about 400°F through a 5-micron filter on leaving the still pot. It will be noted that the metallic impurities, in particular aluminum and silicon, were quite low in the as-received material (Sample 292) and after filtering at 400°F into the hot trap (Sample 293). After hot trapping (Sample 309), small quantities of iron, nickel and silicon were present and attributed to contact with the stainless steel dip leg, thermocouple well, and level probe well during hot trapping. After distilling the first 15 pounds, however, all metallic impurities were below the detection limits (Sample 1031) even though no filter was present between the still receiver and sample tube when this particular sample was taken. A filter was not used because during the first attempt to fill the still pot from the hot trap the upper level indicating "I" probe malfunctioned and hot trapped lithium overflowed into the receiver, requiring that it be returned to the hot trap.⁽⁴⁾ During this transfer the small, stainless steel 5-micron filter on the receiver dip leg plugged and was removed and replaced by a large 5-micron, stainless steel filter after distilling the first 15-pounds of lithium. This 15-pound quantity was distilled in three 5-pound batches and the first two were returned to the hot trap, in order to dilute any impurities which might have been carried into the receiver. The analysis of the residue from the filter is given in Table IV, Sample 1020. The results for Sample 1031; which was taken without a filter, and showed no metallic impurities above the detection limits, indicated that the receiver was no longer contaminated. Nevertheless, it was thought advisable to place a new, and larger filter in the exit line from the receiver.

(4) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 8 for Period Ending April 15, 1967, NASA Contract NAS 3-6474, NASA-CR-72335, p. 33.

Sample 1488, from the charge pot, was analyzed by both GE and NSL with roughly comparable indications of high aluminum, calcium, and silicon. However, GE found 50 ppm of copper while NSL did not detect it at the 5 ppm level. Another sample (No. 1492) was taken after the 500°F loop flush and high values were found by GE for aluminum, calcium and silicon, but the referee analysis by NSL indicated that these impurities were below the detection limits. Finally, Sample 1578 was obtained by draining the lithium still pot since it was expected that any impurities would have been concentrated there during the distillation of 35 pounds of lithium. The concentrations of aluminum, calcium, copper, lead and silicon are not sufficiently high to explain the comparable concentrations of these elements found at one time or another in the distilled lithium.

2. Post-operation Alkali Metal Draining, Sampling, and Analysis

Following the plugging problems which caused loop shutdown, as described in Section A3, a sampling and distillation system was designed and fabricated to gravity drain the potassium from the surge tank in a manner to trap any particulate matter for subsequent analysis. This system, shown schematically in Figure 19, was attached to the loop system at the potassium fill valve (KK). The sample tube consisted of a 36-inch length of 1/2-inch O.D. x 0.020 inch thick wall Type 321 stainless steel tubing and was connected as shown in Figure 19 to assure that the sample tube would be filled during the draining operation. The transfer line connecting the sampler to valve KK and the still pot fill valve was made of 1/2-inch O.D. x 0.060 inch thick wall Type 321 stainless steel tubing.

The still was designed with a "filings shield," such that the top could be cut off without introducing filings into the still. The still pot had a capacity of 4800 cc and the receiver had a capacity of 5100 cc. All valves were welded, bellows sealed, Type 316 stainless steel. All joints were TIG welded and the entire system was helium leak checked at room temperature and hot (400-600°F) according to NSP Specification 03-0013-00-B. No leaks were found in excess of 5×10^{-10} std. cc of air per second.

- Notes: 1. All Joints Welded and Helium Leak Checked
2. All Valves Hoke TY445 or HY473A - Welded Bellows and Positive Return

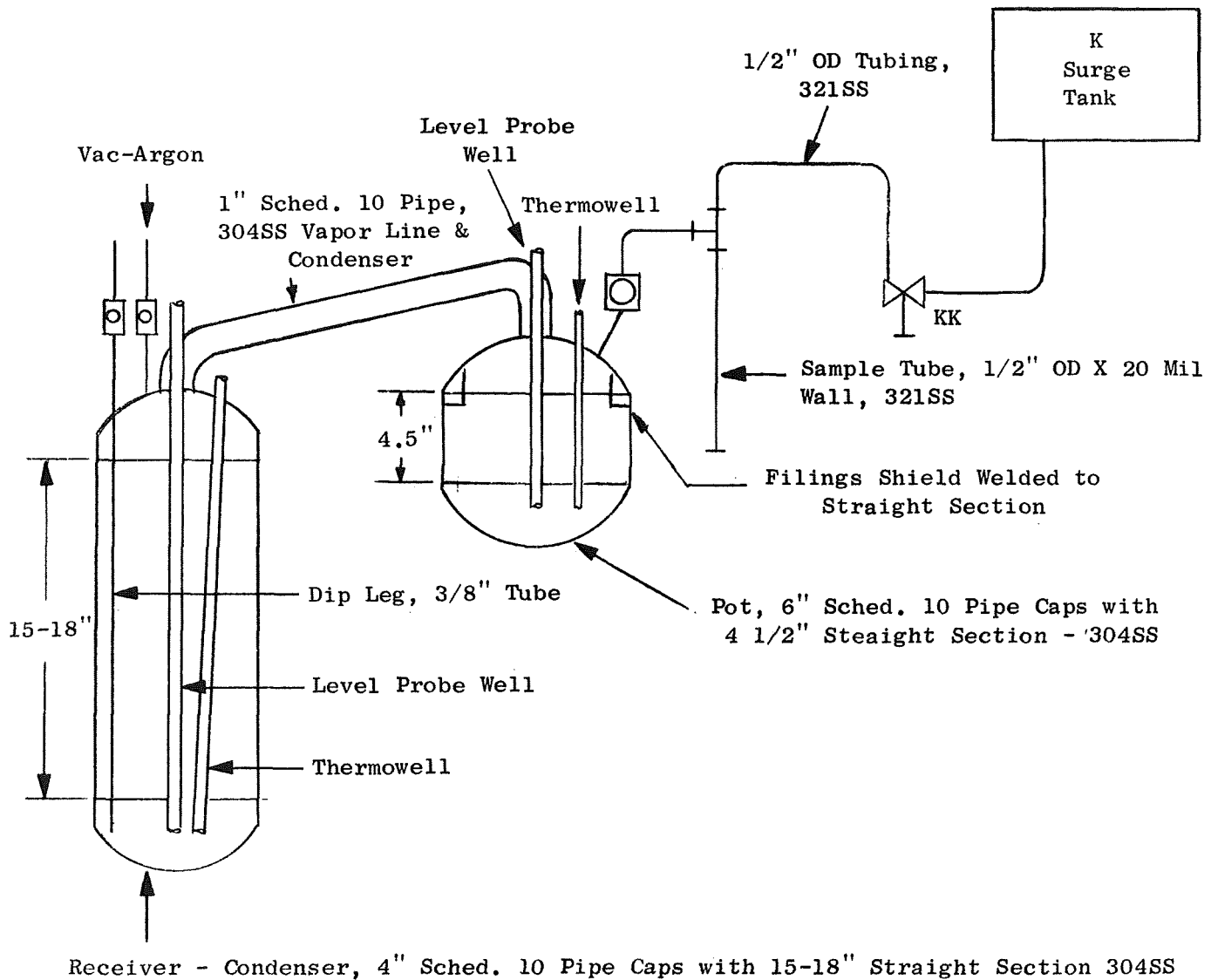


Figure 19. Distillation and Sampling System Used in Isolating Particulate Matter in the Potassium Drained from the T-111 Corrosion Test Loop.

Prior to draining the potassium from the surge tank into the still, the still system was baked out at temperatures from 200° to 600°F until the pressure rise rate was less than one micron-liter per minute. The getter-ion pump on the potassium purification dolly was used to evacuate the system.

The potassium was drained and sampled on April 4, 1968. The potassium surge tank was first pressurized with argon to force the potassium into the loop. The EM pump was turned on and an immediate, but low, potassium flow rate was established in the forward flow direction (The loop was previously shutdown on March 25, 1968 with the potassium loop completely plugged). The loop was immediately dumped into the surge tank and then refilled with a marked improvement in the potassium circulation. The process of filling, circulating, and dumping the loop was repeated five times with a 30 minute dwell time in the surge tank to allow particulate matter to settle out in the surge tank. After the fifth dump, the potassium was drained from the surge tank as described below:

The line from valve KK (see Figure 19) to the loop surge tank was at 350°F to 400°F. The line from the still pot to valve KK was at 200° to 300°F. The drain system was evacuated to less than one micron back to valve KK. Valve KK was cracked and flow began as indicated by a sharp temperature rise in the line between valve KK and the surge tank (from 350° to 450°F) and by the level probe in the still pot. Valve KK was then completely opened and the alkali metal drained from the surge tank until the MSAR inductive type level probe in the still pot indicated 600 to 700 cc of potassium at which time flow ceased. During this time, the sample tube temperature dropped from 300°F to 250°F then increased again to 300°F over a period of about 10 minutes. When flow ceased it was thought that particulate matter might have plugged one of the two valves in the drain system. Therefore the drain system was pressurized with argon up to valve KK to about 27 psia, and valve KK was opened in an attempt to blow argon into the surge tank and dislodge any restricting particles in the line; however, no gas flow occurred as indicated by pressure gages on the surge tank and drain systems. Then valve KK was closed and the drain system was re-evacuated and the surge tank was pressurized to 35 psia and valve KK was reopened. At this point, a small

amount of argon began flowing from the surge tank through the drain system as indicated by the thermocouple pressure gage in the potassium purification dolly vacuum system. While continuously pumping on the drain system the surge tank was repeatedly pressurized until a substantial argon flow was obtained through the drain system; i.e., approximately a 5-psi/minute pressure drop rate in the surge tank. During this period no additional metal flow was detected by the level probe in the still pot. Valve KK was then closed, the drain system was pressurized and the sampler was held under 25 psia pressure at a temperature above the potassium melting point overnight and then cooled to room temperature. All power was shut off on the drain system and all lines from the still pot to valve KK were radiographed to determine if a plug in the lines could be located. The radiographs showed nothing but a few voids in the alkali metal in the sample tube.

The potassium was then distilled from the pot into the receiver for a period of about 66 hours at temperatures of 600° - 700°F. The still pot temperature increased rapidly about 100°F after about 24 hours when the pot boiled dry. The level probe indicated no change in the level of metal in the receiver for a period of at least 16 hours before distillation was stopped.

Following distillation, the potassium drain system and still pot was removed by crimping the line above valve KK, cutting the line between valve KK and the crimp and capping both ends of the cut line with Swageloks. The metal in the line above valve KK had a golden hue and appeared to contain lithium. The entire still-drain system was then placed in the VASCO welding chamber and the sampler and drain line was cut between the still pot and the valve shown in Figure 19, under high purity helium. Both ends of the cut line were Swagelok capped in the chamber. The drain line-sampler system was then removed from the chamber for analysis of the contents of the lines, the results of which will be discussed later.

The still pot was then cut open to investigate any residue remaining in the bottom of the pot. The entire cutting operation was performed in the high purity helium environment of the VASCO welding chamber. The

one-inch vapor line was cut under helium, and a cap was welded on the receiver end to isolate the receiver and its contents. The still pot was cut open with a hacksaw just above the bottom of the filings shield shown in Figure 19. The pot was maintained in an essentially vertical position during the cutting operation which required about eight hours. After the first five hours of cutting the water content of the helium in the weld chamber had increased to 30 ppm. The chamber was evacuated and refilled with dry helium before the final cut off was made. When the top cap of the pot was removed it was noted that the filings shield was intact and therefore no filings from the cutting operation could have fallen into the still pot.

Inspection of the bottom of the still pot revealed black, particulate matter and a thin film of lithium. Most of the black particles were swept into a glass vial using a brush, and the remainder were sucked into an Erlenmeyer flask. The lithium was scraped out and placed in another glass vial and vials were sealed under helium.

A number of specimens were designated as appropriate for analytical chemical, spectrographic or x-ray examination. They are identified and the results are given in Table IV. The significance of the results will be discussed below.

Prior to the final shutdown it was conjectured that the plugging of the metering valve was due to particulate matter and that the boiling instabilities could possibly have been intensified by lithium in the potassium resulting from a leak between the primary and secondary circuits. The lithium transfer line used between valve FF on the transfer system and valve KK on the lithium circuit shown in Figure 20 had been preserved, so a flame photometric analysis was performed on a sample (No. 1683) from this tubing. The lithium was found to contain approximately 200 ppm potassium. The sample (No. 1492) taken after flushing the loop was transferred to the sampler through this line. National Spectrographic Laboratory reported < 25 ppm potassium on this sample (No. 1492). Since lithium and potassium are reported to be essentially insoluble in one another,⁽⁵⁾ such a difference could be due to inhomogeneity in the samples.

⁽⁵⁾ Bohm, B. and Klemm, W., Z. Amorg, Chem., 243, 1939, 69-85.

A potassium sample (No. 1622) comparable to lithium sample 1683 was removed from the potassium transfer line between valves FF and KK. (similar to that shown in Figure 20). The emission spectrographic results shown in Table IV indicate that all metallic impurities except lithium were at or below the detection limits. A flame photometric analysis for lithium indicated 40 ppm. This should be compared with the flame photometric result of <10 ppm lithium for Sample 1487A removed from the charge pot prior to filling the loop; and also with the emission spectrographic result of <2 ppm lithium for Sample 1493B taken after flushing the loop. These results indicate that the leak between the primary and secondary circuits was not present during the initial loop filling and flushing operations.

Potassium Sample 1668A, removed from the bottom third of the sample tube shown in Figure 19, which was obtained during the draining of the potassium loop was also analyzed for lithium by flame photometry. The analysis indicated 8 ppm lithium. Again all other metallic impurities were at or below the detection limits. This sample tube was kept above the potassium melting point overnight after the draining operation to allow any particulate matter present to settle to the bottom. The end cap was removed with a tubing cutter and the potassium dissolved in alcohol. A few black particles were found, but the quantity was insufficient for analysis.

Potassium Sample 1668B was removed from a portion of the heavy walled tubing used as the drain line between valve KK and the still pot (Figure 19). This potassium was flushed from the tubing with distilled water and was converted to the chlorides. A flame photometric analysis of the chloride solution indicated 3.3% lithium. Some black particulate matter was found in the flush water and was analyzed spectrographically, Sample 1668C. This qualitative analysis indicated minor quantities of Cr, Fe, Ni and Ti and trace quantities of Al, Ca, Mg, Mn, Si, Zn and Zr, but no Ta, Hf or W was detected.

Sample 1669A consisted of the black particulate residue which remained in the still pot after distillation. The results of the semi-quantitative emission spectrographic analysis performed by National

Spectrographic Laboratory indicated 25-50% Cr, 25-50% Mn, 15-25% Fe, 5-15% Ni, 5-15% Si, 0.1-0.5% Al, 0.1-0.5% V and <0.05% Ta. These results should be compared with those for Sample 1668B.

Two specimens of this material were prepared for x-ray diffraction analysis. The first, Sample 1669B1, was placed in the glass capillary in an argon filled glove box, while the second, Sample 1669B2 was loaded in air. A best fit analysis of the diffraction data indicated the presence of β -LiFeO₂ and α -LiFe₅O₈. However, after standing in air for a few days another diffraction pattern was obtained which again indicated the presence of lithium iron oxides, as well as Li₂O and α -Fe. The diffraction pattern of the second specimen indicated the presence of lithium, Li₂O, LiOH and α -Fe. No Cr, Mn or Ti was noted for either sample.

Microscopic examination of the particles, Sample 1669A showed them to be agglomerates or curved flakes composed of small particles. Among these agglomerates and flakes, some small silvery white spheres were found which became coated with a chalky white material on standing in air.

A sample (No. 1672) of the metal remaining in the still pot after distillation of the potassium was removed by dissolving in water. The results of flame photometric analyses indicated about 100% lithium and 600 ppm potassium. A flame photometric analysis of the distilled potassium (Sample 1682) indicated 20 ppm lithium.

The conclusions to be drawn from the operations and analyses just presented are as follows:

1. A leak exists in the boiler of the loop between the primary and secondary circuits.
2. The plugging of the metering valve is due to particulate matter which probably originates from stainless steel.
3. The chemical analytical results, to date, do not provide a clear-cut answer to when the leak first appeared.
4. The chemical analytical results, to date, do not provide a clear-cut answer as to the source of the particulate matter although the present indication is that it was introduced from the potassium transfer lines and charge pot.

C. 2600°F LITHIUM LOOP

1. 2600°F Lithium Loop Fabrication

The final assembly of the 2600°F Lithium Loop is scheduled to be accomplished by joining three completely welded and annealed subassemblies: the surge tank subassembly, the EM pump subassembly, and the heater subassembly. The EM pump subassembly fabrication was described previously,⁽⁶⁾ and has now been completed with the welding of the torsion tube to the pump duct. The fabrication of the surge tank subassembly was also completed by welding the Cb-1Zr end caps to the surge tank shell. Both subassemblies were then annealed in furnaces qualified in accordance with Specification SPPS 03-0037-00-A at 2400°F and 2200°F for one hour respectively. Two Cb-1Zr to Type 316 stainless steel bimetallic joints were then welded to the surge tank subassembly. These weldments will be annealed locally at 2200°F for one hour in the vacuum environment of the welding chamber.

The lithium heater subassembly which consists of the heater, the tensile test specimen holder, and the corrosion test specimen holder will be assembled after the selection of the heat treatment specification for the ASTAR alloy tensile and corrosion test specimen by the NASA Program Manager. The lithium heater which contains a 1/8-inch diameter T-111 corrosion test rod insert was assembled by first welding the T-111 electrodes to the tantalum bus bars followed by welding the electrodes to the T-111 heater coils.

2. Lithium Capsule Tests of Tantalum- and Tungsten-Alloy Sheet Specimens

Consideration is being given to the possible inclusion of T-111 (Ta-8W-2Hf, weight percent) and W-30Re-30Mo (atomic percent) specimens in the maximum temperature region of the 2600°F Lithium Loop. The processing history of typical specimens indicated that some contamination of the specimens might occur prior to exposure in the loop, and for this reason it was deemed advisable to evaluate typical specimens in 2600°F isothermal lithium capsule tests of 100 hours duration.

⁽⁶⁾ Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 11, for Period Ending January 15, 1968, NASA Contract NAS 3-6474. NASA-CR-72383

The conditions of the various sheet specimens prior to exposure to lithium are listed below:

T-111, as-received.

T-111, as-received plus autoclaving for 3 hours at 3180°F in 10,000 psig helium plus 3 hours at 2600°F in 200 psig helium.

W-30Re-30Mo, ^(a) powder process product sheet, as-fabricated.

W-30Re-30Mo, Powder process product sheet, as-fabricated plus autoclaving for 3 hours at 3180°F in 10,000 psig helium.

W-30Re-30Mo, arc cast product sheet, as-fabricated.

W-30Re-30Mo, arc cast product sheet, as-fabricated plus autoclaving for 3 hours at 3180°F in 10,000 psig helium.

Two T-111 alloy capsules, measuring 1.0-inch diameter x 0.10-inch wall thickness x 5.5-inch length, were used to test the specimens listed above. The two T-111 alloy sheet specimens were placed in one of these capsules and the four W-30Re-30Mo alloy sheet specimens were placed in the other capsule. Lithium used to partially fill these capsules was purified by hot-gettering with zirconium sheet for 240 hours at 1500°F. Samples of lithium taken during the filling operation were analyzed and found to contain 12 ppm nitrogen. Both the capsule filling and electron beam welding sealing techniques were performed in a 10^{-5} torr vacuum environment. The filling and sealing technique used has been described in detail previously. ⁽⁷⁾

The filled capsules were wrapped with Cb-1Zr foil and heated for 100 hours at 2600°F in a Brew vacuum furnace at a pressure of less than 1×10^{-5} torr. Following completion of the test, the capsules were opened in an inert atmosphere chamber, and the lithium was melted and drained from the capsules. Residual lithium was removed from the tungsten alloy specimens by distillation while the T-111 specimens were cleaned by dissolution of the residual lithium in liquid ammonia. Weight change,

(a) Atomic percent.

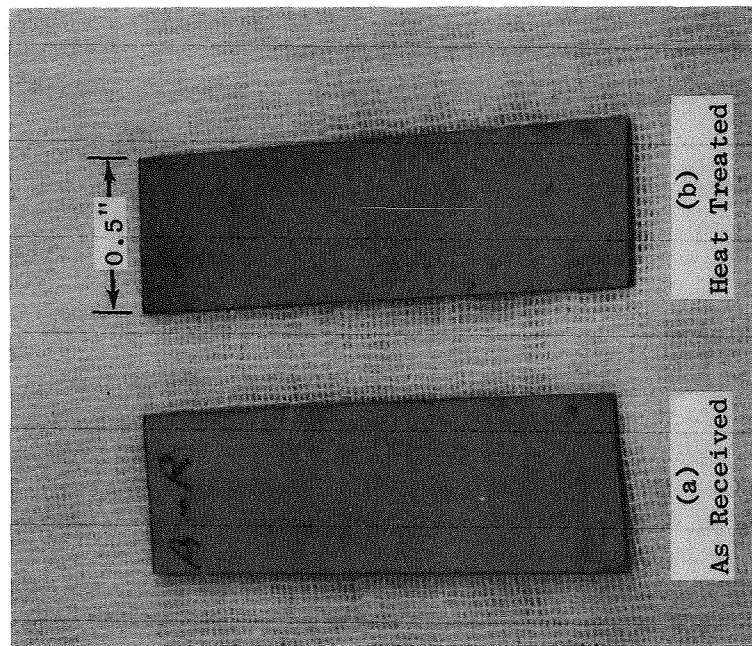
(7) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 11 for Period Ending January 15, 1968, NASA Contract NAS 3-6474, NASA-CR-72383.

chemical analysis, metallographic examination, and microhardness surveys were used in the evaluation of the six test specimens.

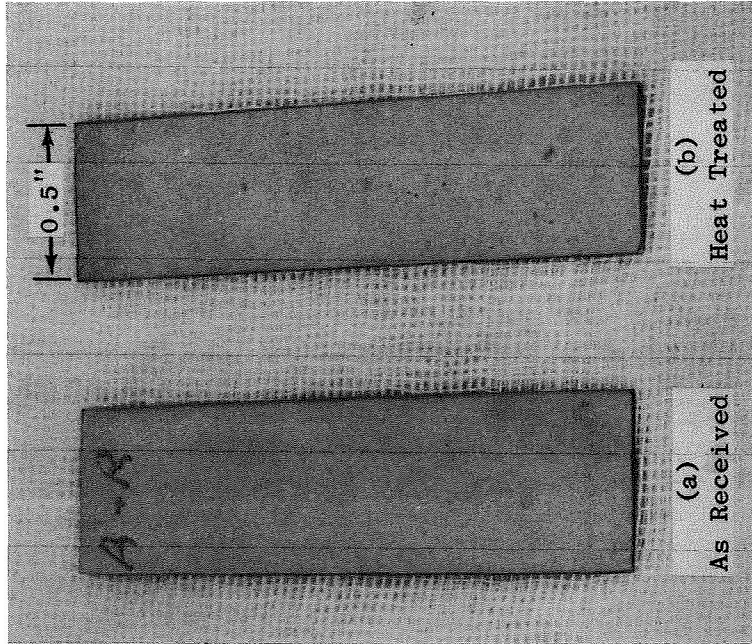
The pretest and posttest appearance of the T-111 sheet specimens is illustrated in Figure 21. Chemical analysis and weight change information on the T-111 specimens is given in Table III. A total of three analyses at two different laboratories were performed for most of the elements listed. The principal changes noted as a result of the 100-hour exposure to the lithium were the significant weight loss and oxygen concentration decrease in the sheet specimen which was autoclaved and heat treated prior to exposure. Approximately 80 percent of the total weight loss noted is attributed to the phenomenal decrease in oxygen concentration. Previous investigators have noted substantial leaching of oxygen from unalloyed columbium and tantalum by lithium⁽⁸⁾ but never with gettered alloys, such as T-111 containing 2% hafnium, to the extent noted in this experiment. The relative high temperature of this experiment compared to most of the other test results which have been reported is thought to be the major factor responsible for the nearly total deoxidation observed in the current experiment.

The metallographic appearance of cross sections of the T-111 sheet specimen which was heat treated before exposure to lithium in a T-111 capsule for 100 hours at 2600°F is shown in Figure 22. The gross oxygen contamination of the T-111 sheet is readily apparent as evidenced by the abundance of both grain boundary and bulk precipitate, which is assumed to be hafnium oxide particles. Lithium exposure caused a substantial reduction in the amount of oxide precipitate in the structure. The surface regions are particularly void of precipitate suggesting that a slight oxygen gradient still exists in the sheet specimen despite the full-section oxygen concentration of only 24 ppm. Diamond Pyramid Hardness (DPH) surveys were performed on specimen cross sections before and after exposure and indicated no detectable hardness gradient in the specimens. It is interesting to note that the sheet specimen was slightly harder following lithium exposure (and deoxidation) than before test. No corrosion of the specimen surface could be detected.

(8) DiStefano, J.R. and Litman, A.P., "Effects of Impurities in Some Refractory Metal - Alkali Metal Systems," Corrosion, December 1964, p. 392t.



Before Lithium Exposure



After Lithium Exposure

Figure 21. T-1111 Alloy Sheet Specimens, as Received and Heat Treated (3 hr at 3180°F in 10,000 psig Helium plus 3 hr at 2600°F in 200 psig Helium), Shown Before and After Exposure to Lithium at 2600°F for 100 Hours.

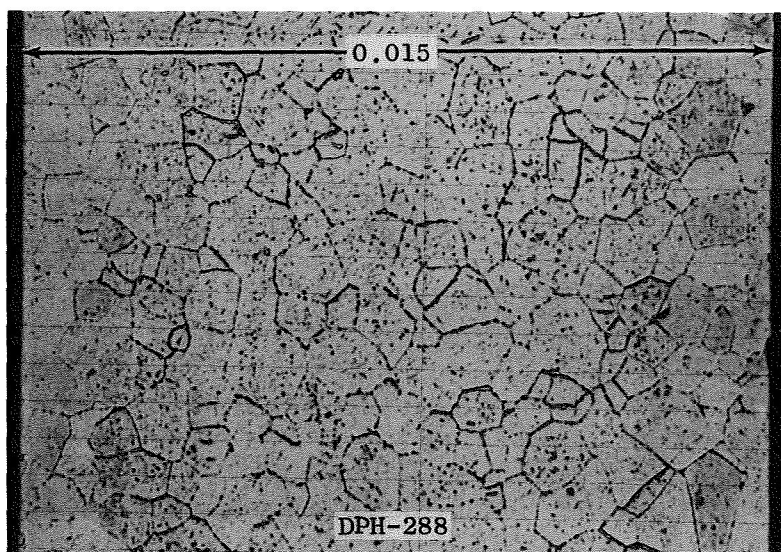
TABLE V.
CHEMICAL ANALYSIS OF T-111 SPECIMENS BEFORE AND FOLLOWING
EXPOSURE TO LITHIUM^(a) FOR 100 HOURS AT 2600°F

Specimen Description ^(b)	Concentration, ppm				Weight Change mg cm ⁻²
	O	N	H	C	
T-111 Sheet, As received Before Lithium Test:	35,35 ^(c) (29)	16,15 (5)	1,1 (< 1)	110,122 (93)	-0.036
After Lithium Test:	18,11	37,44	1,1	158 (87)	
T-111 Sheet, 3 Hours at 3180°F in 10,000 psig Helium Plus 3 Hours at 2600°F in 200 psig Helium . Before Lithium Test:	2877,2164 (2292)	18,15 (19)	20,21		-0.24
After Lithium Test:	24,24 (24)	75.69	2,2	183 (20)	

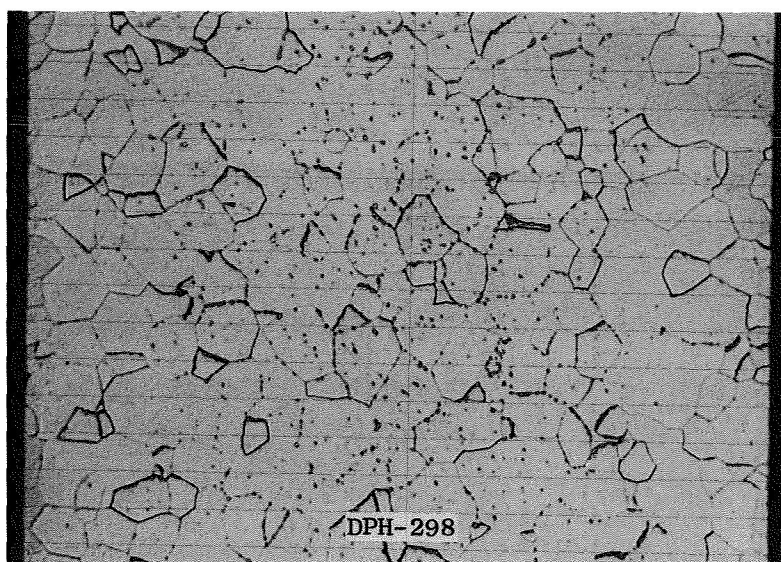
(a) Container capsule: T-111, nitrogen concentration of lithium before test: 12 ppm

(b) Sheet specimen thickness: 0.015-inches

(c) Upper numbers: GE-SPPS analysis; lower number (): GE-NMPO analysis



(a) Before Exposure to Lithium
Oxygen Conc. (avg.) = 2440 ppm



(b) Following Exposure to Lithium
Oxygen Conc.: 24 ppm

Figure 22. Metallographic Appearance of Heat Treated T-111 Sheet Specimens Before (a) and Following (b) Exposure to Lithium for 100 Hours at 2600°F.

Etchant: $30\text{gNH}_4\text{F}-50\text{mlHNO}_3-20\text{mlH}_2\text{O}$

(a) F190313 (250X)

(b) F190413 (250X)

The chemical analyses of the powder product and arc cast product W-30Re-30Mo (a/o) sheet before and following exposure to lithium for 100 hours at 2600°F are given in Table IV. No significant changes in concentrations of oxygen, nitrogen, hydrogen, or carbon were detected. The GE-NMPO carbon analyses, which are considerably lower than the GE-SPPS carbon numbers, are considered to be the more accurate of the two sets of values. Unfortunately, there was insufficient sample to obtain posttest GE-NMPO carbon values. The positive weight changes listed are attributed in large measure to surface contamination during the vacuum distillation operation used to remove residual lithium from the specimens. (Dissolution in liquid ammonia has been adopted as the standard cleaning procedure for alkali metal test specimens.)

The metallographic appearance of the autoclaved W-30Re-30Mo sheet specimens before and following exposure to lithium is shown in Figures 23 and 24. Powder product sheet specimens of the alloy are presented in Figure 23 and show the fine-grained structure typical of the alloy prepared by this method. Examination of the surface of the tested specimen revealed no evidence of corrosion. The grain boundary particles evident in the microstructure of the tested specimens is sigma phase which developed as a result of the thermal exposure of the corrosion test. Microstructures of autoclaved and tested sheet specimens prepared from arc cast alloy are illustrated in Figure 24, and again no evidence of corrosion was observed. The grain size of the arc cast product sheet is quite a bit larger than the powder product materials shown in Figure 23, and the structure is considerably cleaner. Again, a considerable amount of sigma phase developed as a result of the thermal treatment during the corrosion test.

The capsule test results described above indicate that T-111 and W-30Re-30Mo (a/o) specimens of the type described above can be placed in the 2600°F Lithium Loop without fear of catastrophic lithium penetration.

TABLE VI.

CHEMICAL ANALYSIS OF W-30Re-30Mo (a/o) SHEET SPECIMENS BEFORE AND
FOLLOWING EXPOSURE TO LITHIUM^(a) FOR 100 HOURS AT 2600°F

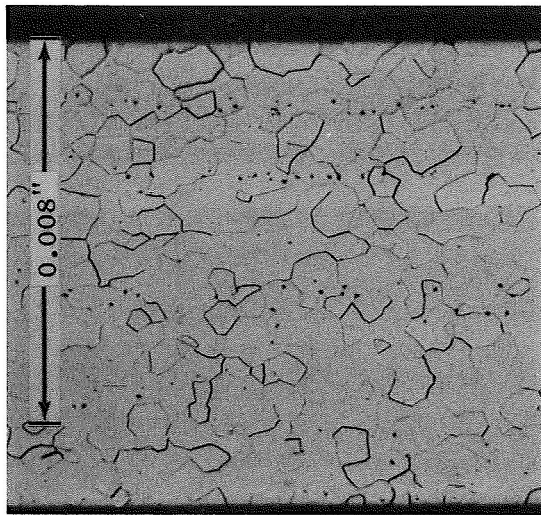
Specimen Description ^(b)	Concentration, ppm				Weight Change mg cm ⁻²
	<u>O</u>	<u>N</u>	<u>H</u>	<u>C</u>	
Powder Product Sheet, Before Autoclaving: ^(c)					
Before Lithium Test:	18,12 ^(d)	1,4	1,1	77,4	
After Lithium Test:	26,18	3,1	2,1	25	+ 0.033
Powder Product Sheet, After Autoclaving:					
Before Lithium Test:	18,9	2,1	1,2	46,13	
After Lithium Test:	46,17	5,1	3,1	23	+ 0.07
Arc Cast Sheet, Before Autoclaving:					
Before Lithium Test:	59,11	5,2	4,1	96,19	
After Lithium Test:	52,27	4,4	3,1	82	+ 0.088
Arc Cast Sheet, After Autoclaving:					
Before Lithium Test:	30,20	6,3	2,2	63,11	
After Lithium Test:	43,26	2	2,1	52	+ 0.10

(a) Container capsule: T-111; nitrogen concentration of lithium before test = 12 ppm

(b) Sheet specimen thickness: 0.010 inches

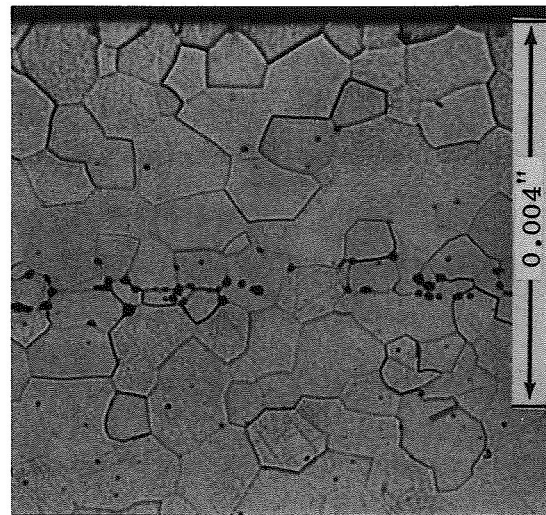
(c) Autoclaving treatment: 3 hours at 3180°F in 10,000 psig helium

(d) First number: GE-SPPS analysis; second number: GE-NMPO analysis



250X

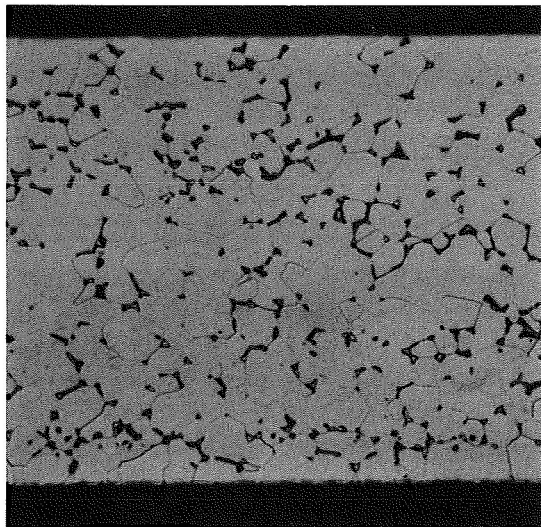
E970311



500X

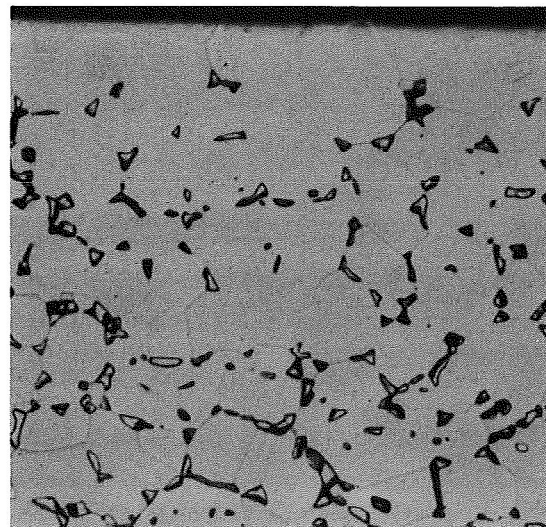
E970312

(a) Before Exposure to Lithium



250X

E970411



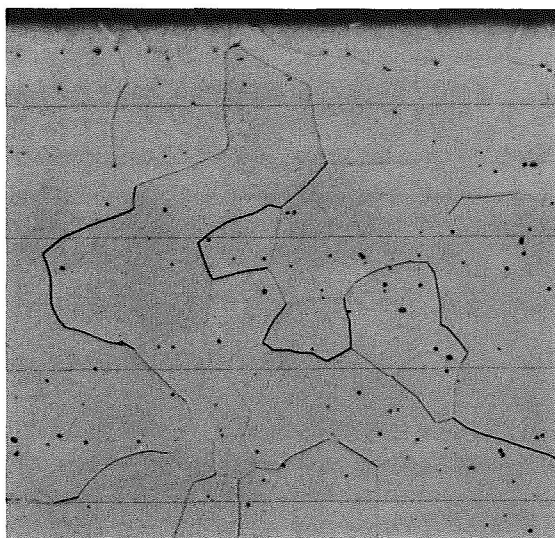
500X

E970413

(b) Following Exposure to Lithium

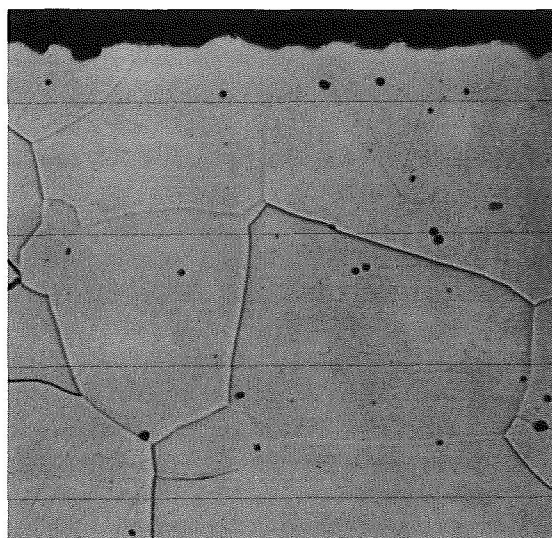
Figure 23. Metallographic Appearance of Heat Treated W-30Re-30Mo(a/o) Powder Product Sheet Before (a) and Following Exposure to Lithium for 100 Hours at 2600°F. The Second Phase in the Microstructures is sigma.

Etchant: 20% Murakamis



250X

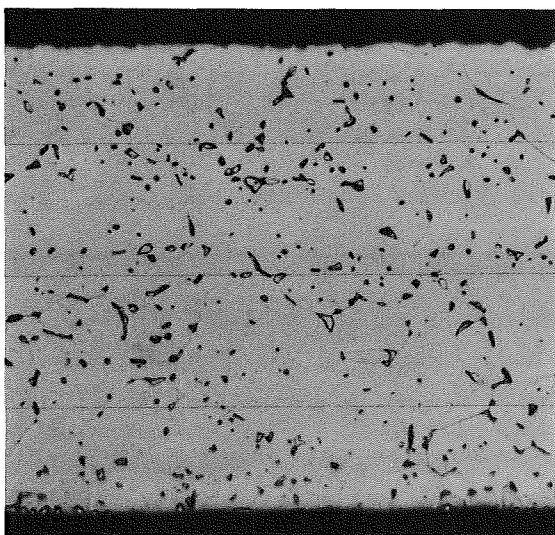
E970711



500X

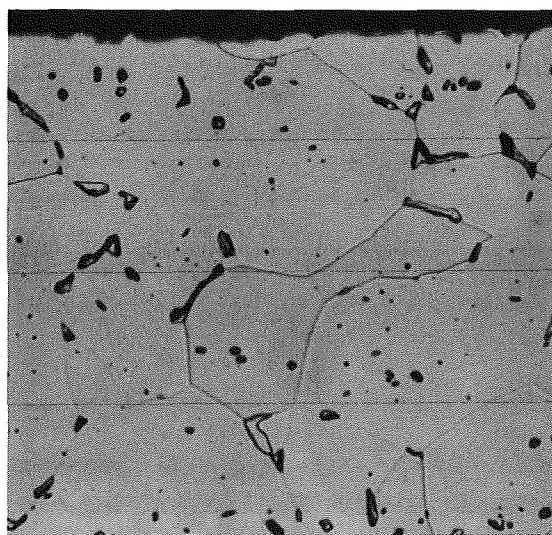
E970712

(a) Before Exposure to Lithium



250X

E970811



500X

E970812

(b) Following Exposure to Lithium

Figure 24. Metallographic Appearance of Heat Treated W-30Re-30Mo(a/o) Arc Cast Product Sheet Before (a) and Following Exposure to Lithium for 100 Hours at 2600°F. The Second Phase in the Microstructures is sigma.

Etchant: 20% Murakamis

IV. FUTURE PLANS

- A. Prepare a test plan for repair of the T-111 Corrosion Test Loop boiler.
- B. Distill the residual alkali metal from the test loop in preparation for repair of the test loop.
- C. Investigate the possible source of the particulate matter observed in sampling the alkali metal after test operation.
- D. Complete the fabrication of the 2600°F Lithium Loop.

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